VACUUM

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in
Vacuum Research and Engineering

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OUR JOURNAL

THE ENTHUSIASTIC RECEPTION OF 'VACUUM' has confirmed our belief that such a journal was needed for the rapidly extending interest in vacuum work. We are gratified that in the short period of four months since the first issue in January 1951 subscriptions have been received from no less than twenty-two different countries.

Our sincere thanks are due to the large number of correspondents who have wished the journal success and who have assisted practically by subscribing and making valuable suggestions for future features. We particularly welcome our readers' views on all aspects of 'Vacuum'.

We regret that for reasons beyond our control the delayed issue of our first number has had its inevitable repercussion upon the timing of the second number, but we shall get into our planned quarterly appearance before the first volume is completed.

TRAINING FOR VACUUM WORK

THE INCREASING COMPLEXITY and extent of technology as scientific discoveries become industrial processes has created a problem in the supply of technically qualified people. As a result, attention has been focussed on technical training in this country and much argument has taken place on the respective merits of university and technological institute. Undoubtedly the latter has made notable contributions to specialised technical training, an outstanding example being chemical engineering, recognised in a recent Government publication as important for our economic development. Nuclear engineering, already taught in established courses in America, and to a much more limited extent in England, is the latest specialist training and likely because of the urgent demand for experts to become speedily established.

It would certainly be over-enthusiasm to suggest that vacuum engineering should be similarly considered, but the principles are of such fundamental importance to so many branches of science and technology that some thought must be given to its teaching. This, admittedly, presents difficulties with already crowded curriculums, but it cannot be left solely to be acquired in the course of professional occupation. Much basic work requires to be done before the design of vacuum plant rests on a less empirical basis and this can only come from teaching ; moreover a knowledge of the subject acquired in student days would undoubtedly engender an earlier appreciation of its usefulness with subsequent advantage to industry.

Vacuum engineering could valuably be taught as an optional course where group studies are selected by the student for his final year, but a very usefnl start has been made at some technical institutes with courses for professionally interested people, and we are glad to publish in this number an account of one such successful course now well established at the Northampton Polytechnic, London.

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Born at Newcastle-on-Tyne in 1907, Professor Tolansky took his first degrees at Newcastle, subsequently continuing with studies in spectroscopy at Berlin and at Imperial College, London. In 1934 he was appointed Assistant Lecturer at Manchester University where he took charge of the Optics side of the post-graduate research work, becoming ultimately Reader in Physics in 1945, and in 1947 was appointed to his present post.

Professor Tolansky's research work has been in two somewhat distantly related branches namely (*a*) hyperfine structure studies in line spectra and atomic nuclear spin, and (*b*) multiple-beam interferometric studies of surfaces and thin films. In these separate fields he has published some 60 papers and 4 books, and was awarded the Boys Prize by the Physical Society of London for his contributions to interferometry. There are in his research laboratory at present more than a dozen post-graduate workers (half of them from abroad) most of whom are actively engaged in the intensive application of the new interference methods for which he is largely responsible, and by which surface features even only of molecular dimensions can be revealed by simple but elegant means. These methods are attracting more and more attention abroad, having wide application in such diverse fields as crystallography, metal physics, crystal oscillations, surface finish of metals, etc. Professor Tolansky will deliver a series of lectures on the subject of Multiple-Beam Interferometry at the Squire Signal Laboratory, Fort Monmouth, New Jersey, U.S.A. on June 28th and 29th next.

In the accompanying article Professor Tolansky gives some highly personal opinions on the relation of vacuum techniques to the techniques of multi-beam interference.

MULTIPLE-BEAM INTERFEROMETRY and the influence of vacuum technique

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Summary

A DISCUSSION of the influence of specific vacuum techniques on the preparation of the highly reflecting thin films used for multiple-beam interferometry. It is shown that the absorption of the film plays an important part particularly with reflection type fringes and that it should be as low as possible. The factors affecting absorption are surveyed. It is concluded that good films are produced by evaporation of silver using a mercury diffusion pumping system with an efficient liquid air trap. Oil (or Silicone) diffusion pumping systems without traps can produce films of high reflectivity but the absorptions of such films are greater than those using the mercury pump with trap. Cleaning techniques and empiric methods for assessing the films formed are described in detail. Silver is the best metal so far available for multiple-beam interferometry.

Sommaire

DISCUSSION DE L'INFLUENCE des techniques du vide particulières sur la préparation des couches minces à haut pouvoir réflecteur utilisées pour l'interferométrie à rayons multiples. Il est montré que l'absorption du film joue un rôle important, surtout avec des franges de réflection, et devrait être maintenue aussi faible que possible. Les facteurs influençant l'absorption sont passés en revue. Il est conclu que de bons films sont produits si l'on utilise une pompe à mercure à piège à air liquide efficace et évaporant de l'argent. Des pompes à huile (ou silicone) peuvent produire d'excellents films, mais leur pouvoir réflecteur est inférieur à ceux produits à l'aide de pompes à mercure avec piège efficace. Des techniques de nettoyage et des méthodes empiriques pour évaluer le depôt sont décrites en détail. Pour l'instant, l'argent est le meilleur métal par l'interférométrie a rayons multiples.

INTRODUCTION

IT IS the purpose of this article to discuss the importance of the vacuum technique involved in the production of the highly reflecting thin films which are required for an adequate resolution in multiple-beam interferometry, particularly that aspect concerned with the study of surface topography. It will be necessary to survey first in a brief way the principles of multiple-beam interferometry, but only in so far as the properties of the vacuum-deposited thin film are involved.

Within the past few years there has been a considerable outburst in activity in the study of the reflecting and optical properties of thin films both metal and dielectric. The formidable recent bibliography has been admirably collected and classified by P. Cotton and P. Rouard in the Proceedings of the Conference on the Optical Properties of Solid Thin Films (held in Marseilles in April 1949 ; proceedings published in France in 1950 by the C.N.R.S. Journal de Physique et le Radium, Vol. 11, July 1950) and as this contains details of 250 papers, mostly recent, very few references will be given here. The discussion will largely be concerned with the personal views of the author and will be strictly limited to the aim outlined above.

* M.S. received April, 1951.

PRINCIPLES OF MULTIPLE-BEAM INTERFEROMETRY

The term " Multiple-beam Interferometry " is now often used specifically to describe the methods of studying surface topography by the use of various kinds of interference fringes, wherein for each case the incident light falls onto a high reflecting, partially transmitting thin film of metal and then meets another

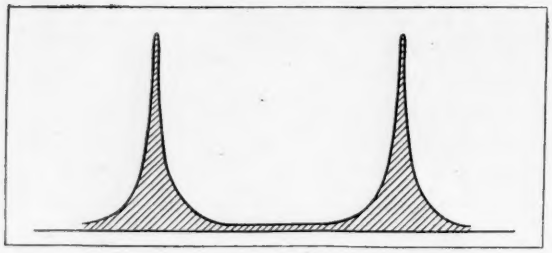

Fig. 1.—Schematic diagram of the shape of transmission fringes for reflectivities >0.8.

closely placed high reflecting film. The light beams are reflected to and fro between the optical surfaces producing the interference effect with the result that sharp fringes are produced. There are other variants which use this or analogous optical arrangements. For example the Fabry-Perot interferometer is essentially a multiple-beam, interference instrument. Indeed the discussion here regarding topographical studies of surfaces and studies of thin films holds largely also for the Fabry-Perot interferometer, so much so that it is simpler to approach the theory of fringe sharpening from this more familiar instrument.

It will become evident later that two separate (but related) cases exist in practice, namely those in which fringes are viewed respectively in transmission and in reflection and it will be established that, apart from basic intrinsic optical factors, the accuracy and precision of the techniques depend largely on two properties of the reflecting films and these are (*a*) the reflectivity, (*b*) the absorption. Both are highly sensitive to the vacuum techniques used.

The Fabry-Perot interferometer has been actively employed in spectroscopy and metrology for 50 years, and attention has almost uniquely been paid only to the reflectivity of the metal films used. It is only within recent years that the importance of *absorption* in multiple-beam interferometry has been recognised (see S. Tolansky, Multiple-Beam Interferometry of Surfaces and Films, Clarendon Press, Oxford, 1948, and High Resolution Spectroscopy, Methuen, 1946).

When discussing the theory of the multiple-beam interference of the Fabry-Perot interferometer, most optics books, whether elementary or advanced, neglect the absorption and derive the fringe shape for both the cases of transmission and of reflection for the non-existent ideal case of a thin metal film without absorption. In fact absorption has a most important influence and it will be demonstrated later that this question is very closely tied up with the vacuum practice adopted.

TRANSMISSION FRINGES

It is well known that the deposition of a reflecting film on the two surfaces of an interferometer leads to a fringe sharpening in accordance with the familiar Airy sum formula such that the intensity distribution for a beam of unit incident intensity is given by :—

$$I = \frac{T^2}{(1-R^2)} \times \frac{1}{1+(4R/(1-R)^2)\sin^2 \delta/2}$$

in which R and T are the reflecting and transmission coefficients and δ is the phase difference between the successive multiple-reflected beams. The fringes have a shape depending only on the value of R and vary from a maximum value $T^2(1-R)^2$ to a minimum value $(1-R)^2/(1+R)^2$. As is well known, when R is high, (say > 0.8), then the fringe shape of the type schematically shown in Fig. 1 results, and the fringe sharpness permits accurate measurement. This condition can be made to apply effectively to the localized multiple-beam wedge fringes used for topography as well as to the Fabry-Perot interferometer, providing certain critical optical conditions are fulfilled. The half width of such a fringe, as a fraction of an order is $(1-R)/\pi\sqrt{R}$ which has the respective values 1/14, 1/19, 1/30, 1/50 for R values respectively 0.80, 0.85, 0.90, 0.94.

[It is difficult to obtain R > 0.94 for the green light which is frequently used.]

One therefore aims at a high R value to produce as narrow a fringe as possible. Since the *minimum* is numerically not very different from $(1-R)^2/4$ and as R is selected as close as possible to 1 this minimum is normally sufficiently slight to be treated as zero for our purpose, although it is of some concern in the

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Fabry-Perot interferometer.

It will be noted that the assumption that there is no absorption means that $1 - R = T$, thus one arrives at the somewhat unexpected conclusion that the intensity of the fringe maximum $\left[i.e. \dfrac{T^2}{(1-R)^2} \right]$ is always unity, *i.e.* equal to that of the incident light no matter what the value of the reflecting coefficient, and despite the throwing back of the major portion of the incident light at the first reflecting surface. In fact this condition does not exist, for the metal film always absorbs and one must write $1 = A + T + R$ in which A is the absorbed fraction. Taking the simplified case in which both metal films of the interferometer are identical this leads to :—

$$I_{max} = \left(1 + \frac{1}{A/T}\right)^2 \text{ or } \left(\frac{1-R-A}{1-R}\right)^2$$

$$\text{and } I_{min} = \left(\frac{1-R-A}{1+R}\right)^2$$

The whole scale is thus reduced by the factor

$$\left(1 + \frac{1}{A/T}\right)^2$$

but the ratio I_{max}/I_{min} remains as before

$$\left(\frac{1+R}{1-R}\right)^2.$$

The effect of absorption is therefore to reduce the total intensity without affecting the shape.

To show how serious this can be, Fig. 2 gives the calculated fall off in the intensity of the fringe maxima for various reflectivities, if one postulates a uniform absorption of only 5% for films with reflectivity >0.75 (this is in fact a typical value). The intensity falls with great rapidity as R increases, becoming zero at R = 0.95. Thus whilst an increase in R from 0.90 to 0.94 reduces line half-width from 1/30th to 1/50th of an order, the intensity in practice can be reduced from 1/4th of the incident light to less than 1/36th. Such a severe reduction in light cannot always be tolerated, especially if a surface is under microscopic examination, even only with medium powers. Since absorption conditions happen to be very critical in the higher reflecting ranges, the reduction in intensity may in fact far exceed the above and in individual cases an intensity reduction factor of many *hundreds* might well (and often is) involved.

This discussion shows that in the deposition of metal films for interferometry using fringes in *transmission* the absorption of the film is at least as important as the reflectivity and this aspect will receive much discussion later when dealing with experimental vacuum techniques. Clearly if absorption is lowered one can use increased reflectivities with improved definition and vice versa.

REFLECTION FRINGES

Until the writer drew attention to the position, the profound effect of absorption upon reflection fringes had gone unnoticed. Fringes in reflection are now widely used, especially since interferometry is being applied to measure the thickness of thin films and to the study of metals, including polished, worked and deformed surfaces, single crystals, etc. The

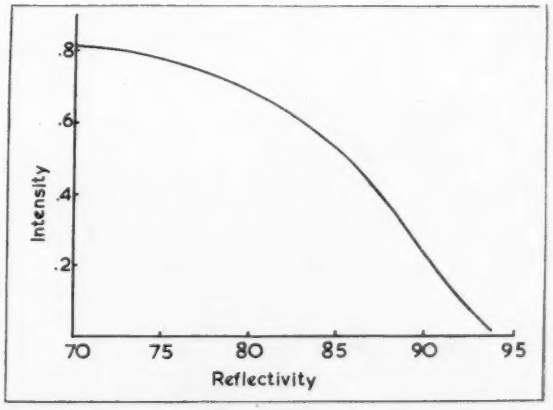

Fig. 2.—(Calculated) Intensity of the fringe maxima for various reflectivities >75% assuming uniform absorption of 5%.

effect of absorption on transmission fringes is merely to reduce the overall intensity, but the effect of absorption in the reflected system is profound, for it can well lead to effective obliteration of the whole fringe system and in fact the failure to appreciate this position was responsible for a lack of development for many years.

Elementary theory shows that with no absorption reflected fringes are exactly complementary to the transmitted system and appear as fine dark lines on a bright background, if high reflectivities are employed. In fact, one need only invert Fig. 1 to give the predicted distribution which is shown in Fig. 3. The maximum is $\frac{4R}{(1+R)^2}$, which is usually almost 1, being 0.98 even for the low value of $R = 0.75$. The *minimum is always zero* quite independently of the value of R. According to this idealized argument the reflected fringes are always completely

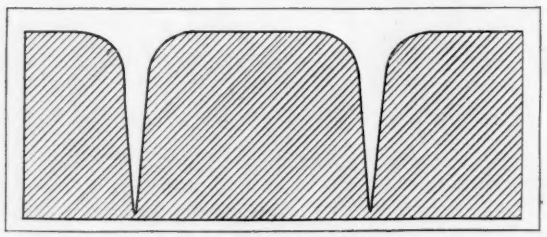

Fig. 3.—Schematic diagram of the shape of reflection fringes assuming there is no absorption.

black and therefore visibility is very high. But if account is taken of the absorption, the whole position changes radically. The only absorption factor involved in the calculation is that of the *first* surface, *i.e.* that nearer the light source. If the absorption term A is introduced then the maximum becomes

$$I'_{max} = \frac{4R}{(1+R)^2}\left(1 - A + \frac{A^2}{4}\right)$$

and as A is often not much more than 0.05 effectively, I'_{max} is not affected seriously and the maxima are only a few per cent. less than those calculated for zero absorption. However, the *minima* (formerly zero) are quite differently affected and instead of zero have the new value $I_{min} = R\left(\frac{A}{1-R}\right)^2$. Again taking $A = 0.05$, this gives for different reflectivities :—

R	= 0.75	0.80	0.85	0.90	0.94
I_{min}	= 0.03	0.05	0.09	0.22	0.65

Thus for $R > 0.90$ the minimum rises with great rapidity, becoming in fact equal to the maximum at $R = 0.95$, which means that no fringes are visible at all, but long before this they are effectively invisible.

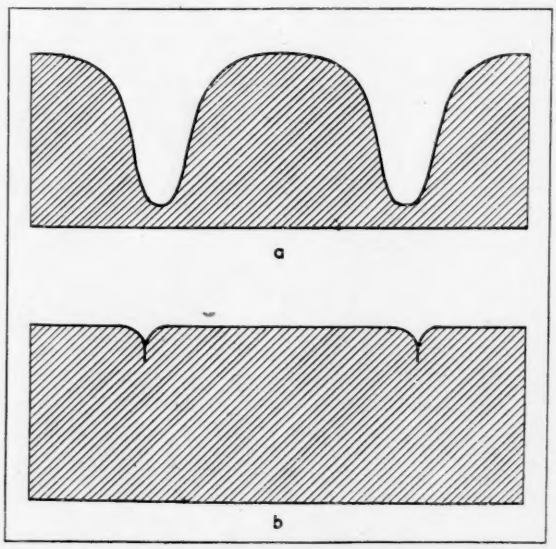

Fig. 4.—Schematic diagram showing the effect of reflectivity on the quality of the fringes :—(*a*) low reflectivity—broad fringes (*b*) high reflectivity—sharp fringes.

As before, the fringe width decreases with increasing R so that in effect the fringes become sharper and sharper, but less and less visible as R increases. Schematically as shown in Fig. 4a low reflectivity gives broad fringes, easily seen since they are very dark, whereas, in Fig. 4b, high reflectivity gives sharp fringes, *but they are almost invisible.* Any marked

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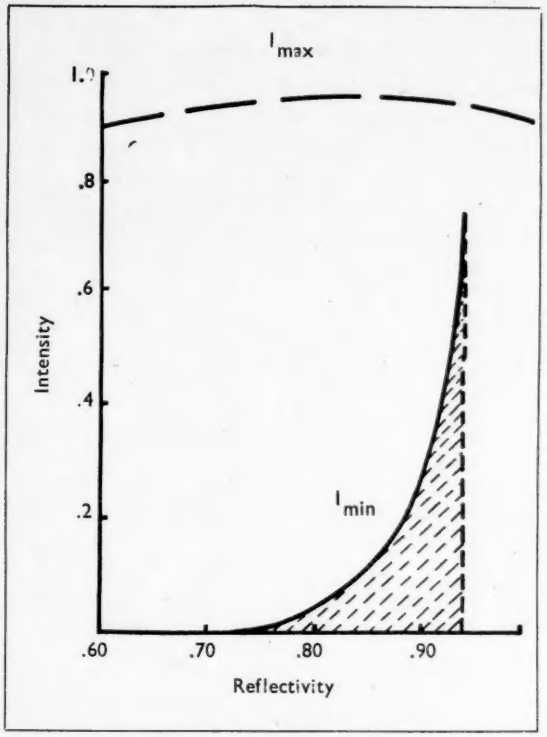

Fig. 5.—Graph showing the calculated distribution of reflection maxima and minima for silver films deposited by evaporation *in vacuo*.

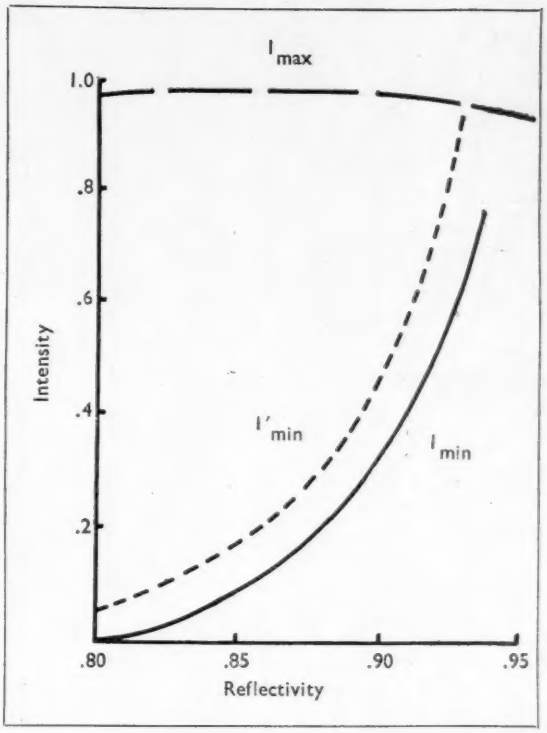

Fig. 6.—The influence of absorption on the intensity minima of fringes.

increase in A has a catastrophic influence in the region of higher reflectivity. Thus with reflection fringes the more important factor is not high reflectivity, but *low absorption*.

Fig. 5 gives the calculated distribution of reflection maxima and minima for silver films deposited by thermal evaporation using an oil diffusion pump. The upper flat curve shows that the maxima for R > 0.70 are practically constant, being some 94% intensity of the incident light. The intensities of the *minima* can be imagined by dropping down from the maxima to the lower curve. It is as if the dark fringe were diluted by the light represented below the lower curve, *i.e.* the shaded area. With the higher reflectivities this dilution is very severe and the visibility falls correspondingly. The curve is based on some experimental values of the absorption A which increase regularly from 0.03 at reflectivity 0.70 to 0.053 at reflectivity 0.94. As already indicated a small increase in absorption has a most serious influence and in Fig. 6 the effect on the minima produced by increasing the absorption A merely by 0.01 is to lead to the dotted curve I'_{min}. As a result there is practically complete loss of fringe visibility for R > 0.90.

VARIETIES OF DEPOSIT

Up to within quite recent times high reflecting deposits have invariably consisted of metal films only, but there is a distinct possibility that in the near future one of the interferometer plates at least will be coated

with a composite 'sandwich' film of dielectrics of high and low refractive indices alternating. There are reasons why this new development will have restricted application and the discussion in the first instance will be concerned with metals. Hence there are two variables, (*a*) the choice of metal, (*b*) the method of putting down the film. It is clear that the solid bulk metal must have an inherently high reflectivity and the most suitable yet found is silver (the chemically active alkali metals might in theory have better optical properties, but of course they are quite unusable). The reflectivity of a thick (solid) evaporated layer of silver reaches 0.98 at the wavelength $\lambda = 7000$ Å, and only falls to perhaps 0.90 at $\lambda = 4000$ Å. It then falls steadily and rapidly to perhaps 0.04 in the neighbourhood of $\lambda = 3000$ Å. For interferometry it can therefore only be used for wavelengths exceeding $\lambda = 4000$ Å. Aluminium and magnesium-aluminium alloys have been strongly advocated in some quarters. Aluminium has consistently lower reflectivities than silver in the regions above $\lambda = 4000$ Å, having a fairly uniform reflectivity of about 0.895, and this value is maintained in the ultraviolet to about $\lambda = 2500$ Å. There is some evidence that an aluminium-magnesium combination of composition 10 : 1 may have reflectivity approaching 0.94 over the visible region. For topographical interferometry there is every reason to choose silver from the reflectivity point of view, particularly because one has available as suitable sources either the intense green mercury line or the strong sodium yellow doublet. There are no advantages in working in the red region, for although the slight gain in reflectivity leads to somewhat sharper fringes, on the other hand the separation between red fringes is a proportionably greater distance in angstrom units. It might appear at first that if high reflectivity in the blue, or even ultraviolet, could be secured, that it would indeed be profitable to work with shorter waves since there is then a proportionate gain in fringe separation. But this is paradoxically not so, for in fact topographical studies are most frequently referred to a standard optically worked surface, usually optically worked with green mercury light. A surface correct to $\lambda/100$ at 5000 Å is only correct to $\lambda/50$ at 2500 Å, in other words the defects of the surface exactly compensate the gain in fringe separation and since the ultraviolet involves other complications, even if high reflectivity were available, it would be entirely undesirable to work there. Thus from the viewpoint of reflectivity, sources available, convenience and accuracy, silver, to be used in the green or yellow, is the best medium available.

It will be shown later that apart from these issues, aluminium is to be strongly condemned on grounds of high absorption too. The earlier advocated metal platinum is in a lower class altogether, both as regards reflectivity and absorption and will not be taken into consideration.

However it is not the solid bulk metal reflectivity that matters, but the reflectivity of a partially transmitting thin film and fortunately in this respect silver has excellent characteristics if a correctly made film is used. It is here that the mode of preparation is of paramount importance.

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TYPES OF SILVER FILM

Three different methods have been evolved at successive periods during the past 50 years for the preparation of silver films for interferometry. These are (*a*) chemical deposition, (*b*) cathodic sputtering, (*c*) thermal vacuum evaporation. Although it was the basis of the earlier classical work, the chemical method is now abandoned. Reflectivities are somewhat lower for chemically deposited bulk metal than that given by other methods, but not violently so, varying from 0.95 at $\lambda = 7000$ Å to about 0.86 at $\lambda = 4000$ Å. But for thin films the *absorption* is much worse (it is perhaps not widely known that the best chemically deposited silver films are obtained by deposition *upwards*, i.e. the receiving surface should be placed horizontally facing downwards in the bath). The thin films have a somewhat greyish tint and this is associated with the higher absorption characteristics.

Until about 1935 cathodic sputtering was the recognised best method although silver evaporation for interferometry was successfully used by Ritschl during 1930.

I have had some considerable experience of both

methods and although there is no doubt that very high reflectivities are obtainable by both, it is my belief that the lower absorptions are easier to be obtained in the case of evaporation. The methods will now be compared.

CATHODIC SPUTTERING

When a direct current electric discharge is passed through a gas at a pressure of say 10 to 0.01 mm. Hg, the metal of the cathode, if suitable, sputters and deposits as a mirror on surfaces close to the cathode. It has been established that the mechanism involves the throwing off of neutral particles through the bombardment of the cathode by the positive ions in the gas discharge. There is a good deal of evidence to show that the sputtered metal is in the form of a neutral gas vapour which leaves the cathode with thermal velocities approaching that of the boiling point of the metal, in fact there appears to be a local thermal evaporation. The rate of sputtering is different for various metals and is shown roughly in accordance with the following scheme, in which rates are shown below the metals, for deposition in hydrogen gas.

Bi	Ag	Au	Cu	Fe	Al	Mg
170	80	50	31	7	3	1

The sputtering of Al and Mg is not a practical proposition and the difficulty is attributed to the formation of protective oxide coatings. Silver has ideal sputtering characteristics.

It has been established that the sputtering rate increases with the atomic weight of the gas employed and is for example at least 16 times more effective in argon than in hydrogen, whilst helium has an abnormally low efficiency. This increase in efficiency with mass is due to the more effective bombardment of the cathode by the heavier ions I have found too that there is an improvement both in the rate of deposition and in quality of the film formed by using a liquid air cooled trap. The mechanism is probably that the removal of impurity reduces the possibility of secondary collisions which would adversely influence the rate of deposition and at the same time helps to keep down the absorption of the film by lessening the chances of adsorbed film contamination.

In producing the film the cathode should be a disc of pure silver somewhat larger than the surface receiving the film. A good gas pressure is that which leads to a cathode dark space of some 2 cm. using a d.c. voltage of perhaps some 2000 or more volts. The mean free path under such conditions leads to a rapid fall off in the deposit as the receiving surface is removed away from the cathode. It is best placed no more than 1 cm. beyond the dark space. Under clean conditions a mirror of suitable thickness can be produced in about 10 minutes. The receiving surface is best shielded from the discharge for the first few minutes.

Although vacuum pressure is flexible over a wide range there are two most essential conditions for success and these are that the gas discharge must be free from organic impurities and the receiving surface must be clean. Organic impurities from vacuum greases and seals or from inadvertent use of organic grease-cleansing agents (benzene, for example) can have a catastrophic influence, leading to the formation of dark brown, almost opaque films, entirely useless for the original purpose. Furthermore the organic impurity persists for a long time and may even lead to the abandonment of the plant if the cathode becomes badly contaminated.

I am informed by A. S. D. Barrett that Edwards have used a flushing technique in the production of sputtered mirrors. Instead of maintaining the pressure constant with the usual rotary pump generally used in sputtering work, a high speed diffusion pump is installed pumping against a constant leak of gas to maintain the correct pressure. By this means uniformity of atmosphere is obtained and irregularities due to leakage and gas vapours are- overcome.

Unless the receiving surface is well cleaned local patches with different absorptions appear. Cleaning techniques in general will be discussed later.

One major drawback to cathodic sputtering is that the receiving surface usually heats up, certainly often such that it may be hot to touch immediately after

deposition. This has two disadvantages. First it restricts the method to surfaces which will not be affected by the heating and this is a very serious restriction, especially in the fine scale study of crystal and metal surfaces. Second, time is consumed in permitting the deposited silver to cool down before admitting air into the plant for removal of the film. It must be emphasized that the heating restriction prevents any attempts to sputter on many of the objects which have already been interferometrically studied using evaporated films, *e.g.* low melting point metals, hydrated crystals, plastics, etc. The heating and ionic bombardment can have permanent effects on glass or quartz optical flats. If such surfaces, having been subject to sputter, were not initially quite clean, the sputtered film often shows irregularly shaped patches of different absorptions. These patches are at times 'burnt' into the surface and cannot be removed (drastic polishing and removal of surface material is not of course envisaged). I have resilvered such a surface again and again and always the same pattern has appeared suggesting that surface texture has been affected in a permanent way.

In sputtering a silver mirror, all the exposed metal parts within the vacuum system should be either of silver or of aluminium. Brass and iron are to be avoided since both easily sputter off heavily absorbing materials. A convenient sputtering current is some 20 milliamps.

Although I have personally not made direct measurements of the absorptions of sputtered silver films, intensity and other considerations have led me to the conclusion that the absorption is greater than for the case of evaporated films and I have therefore abandoned sputtering in favour of evaporation. This conclusion is supported upon two other grounds. In the first case I have evaluated the absorptions for a sputtered film from data published by Fabry in 1928 and this leads to a much poorer efficiency than the evaporated films. Secondly it has been my practice to assess films for interferometry by an image counting test which depends partly on the absorption and this has shown me that the sputtered films which I have been able to make are inferior to the evaporated films for interferometry.

THE IMAGE COUNT ASSESSMENT

This method of assessing interferometer mirrors was shown to me by Ritschl in 1931 ; I am unaware as to the identity of the originator. When silver deposition is undertaken two pieces of glass are placed alongside the receiving surfaces and are coated effectively with films identical with that on these surfaces. The two pieces of glass are held in the hands before the eye with silvered surfaces opposing, separated perhaps 1 mm. and slightly inclined to each other. A bright light (pointolite, carbon arc, or bright filament lamp) is viewed from a distance of about 1 metre and a succession of multiple images is seen. These images can be counted. They exhibit a number of characteristics. The first few images are a deep blue but the higher order images become more and more yellow and finally almost red. The higher the reflectivity the slower the fall off in image intensity and the more one can see. For similar mirrors the intensity of the $(n + 1)$th image is $T^2 R^2$ which can be written $(R^n (1 - R - A))^2$. When R is high the transmission T depends very much on the value of A. As a result of this relationship the count turns out to be surprisingly sensitive to R and A. Experience shows that with good mirrors 60 images can be counted. With very good mirrors even 80 and on the rarest of occasions as many as 100 images are seen (the test of good mirrors is their ultimate performance in an interferometer, *i.e.* the fringe sharpness and the brilliancy). With a uniform absorption of 0.05 the relative intensities I_{61} of the 61st images are :—

R	0.94	0.90	0.85	0.80	0.75
I_{61}	1	10^{-1}	10^{-3}	10^{-6}	10^{-9}

The fall-off is extremely rapid and is primarily determined by R but is also affected by A. The combined effect of slow change in colour and long extended count together offer a most valuable empirical rapid method for evaluating the suitability of the film, in a qualitative manner.

I have found in general that the count for a good

sputtered film is less than that for a good evaporated film. When taken in conjunction with Fabry's measurements this implies both lower reflectivity and higher absorption. The rapid convenience of the count is such that other methods of estimating the properties can well be disregarded.

Much space has been devoted here to sputtering and the reason is that there is on record a claim made by Romanowa, Rubzow and Pokrowsky (Phys. Zeit. Soviet, **5**, 1934, 759) in which sputtered films were stated to be produced with a reflectivity of 0.95 at $\lambda = 5500$ Å, a transmission of 0.04 and an absorption of only 0.01. Such mirrors would be more efficient than any yet so far reported by other observers. These authors state that the key factor in obtaining these phenomenal values is purity. The sputtering was carried out in an all-glass equipment, a bell jar on a glass base. Although the cathode was of silver an aluminium anode was used. The equipment was first degassed by hard pumping with a diffusion pump. After this it was flooded with pure hydrogen produced by electrolysis. The surface to be covered was protected with aluminium foil and this was only removed after the discharge had run for half an hour. Gas pressure during sputtering was 0.05 mm. and current 20 milliamps. To reduce heating the sputtered film was made in a succession of separate deposits. The slightest trace of oxygen in the system increases the absorption.

This claim has never been substantiated by other workers but on the other hand, other workers do not seem to have taken the same purity precautions. The procedure should be repeated by others and it is hoped it will be.

PROPERTIES OF EVAPORATED FILMS

It is my view that there is a distinct difference between those silver films evaporated *in vacuo* using oil diffusion pumps and those produced by mercury diffusion pumps which have adequate liquid air traps, and evidence will be produced to substantiate this opinion. From experience gained with both systems, I am satisfied that the use of a mercury pump with adequate liquid air traps can produce films with absorption characteristics better than those films made either with oil or with silicone pumps. For interferometer films there is little significant difference in the *reflectivities* available by these different systems, but in the important feature of *absorption* the mercury pump with liquid air traps appears to be superior. With the mercury system the image count that I have obtained was frequently between 60 and 80, and at times exceeded this, even reaching 100. With oil pumps a count exceeding 60 is infrequent and I cannot recall a count greater than 80, although I have seen many hundreds of mirrors. On empiric grounds alone then the mercury mirrors are clearly superior with regard to reflection and transmission.

For more precise comparative data, three sets of figures for silver are given below, (I), (II) and (III). Those in column I are taken from a paper by Kuhn and Wilson (Proc. Phys. Soc., **63**, 1950, 745) who used a mercury pump with trap ; column II shows some figures I obtained with apiezon oil diffusion pump in 1946 (Physica, **12**, 1946, 649) and III gives measurements made in my laboratory with a large silicone oil diffusion pump by Avery (Ph.D. thesis, London, 1950). Avery has also measured reflectivities and absorptions for evaporated aluminium and these are included under IV for later discussion.

Reflectivity	Silver Absorption A%			Aluminium Absorption A%
R %	I	II	III	IV
	Mercury	Apiezon Oil	Silicone Oil	Silicone Oil
70	—	3	8.5	20
75	5	3	8	19
80	4.8	3.5	8	17
85	3.2	4.5	7.5	13
90	2.6	5.5	6.5	10
94	2.1	5.3	6	—
98	1	—	—	—

More specific experimental details will be discussed

later. The figures have considerable significance and reveal the great variability in the reported data for silver films. These are in any case mean values from numerous films and there is in each case an appreciable scatter of results. Reproducibility is in fact very hard to achieve even for any single apparatus.

The mercury films (I) are best with regard to absorption for reflectivities above 85%, but are inferior to (II) in the reflectivity range 70 to 85%. But the reflectivity >85% is the important region and so striking are the mercury pump figures that Kuhn and Wilson have actually reported the production of mirrors of 98% reflectivity and 1% absorption. It must be pointed out that these reflection figures are quite unusual and rarely has such reflectivity been claimed, although the correspondingly low 1% absorption has been claimed by other workers.

The film (III) is considerably worse than either (I) or (II). It will be recognised too that aluminium is in a much lower class altogether and for this reason, the fringe quality using aluminium is always much inferior than that using silver (except of course in the ultra-violet regions of the spectrum which is an aspect which has been deliberately excluded here).

These definite absorption and reflection figures entirely confirm the qualitative indications of the image counts.

CLEANING TECHNIQUES

The production of the correct type of film involves two aspects of vacuum technique namely (*a*) the surface cleaning operation, (*b*) the vacuum deposition procedure. Surface cleaning is of vital importance in two respects, for not only are the optical properties of the film critically affected by any impurity surface layers on the receiving surface, but also the tenacity of attachment to this surface is also determined by this and with it sensitivity to scratching and damage. From the viewpoint of interferometry there are two entirely distinct cleaning procedures, according to the nature of the surface to be studied. For optical flats of glass or of quartz, or for hard resistant crystal surfaces such as diamond, beryl, topaz etc., one type of cleaning can be adopted, but for other surfaces, *e.g.* metals, or say mica, or plastics etc. quite a different approach is required. The cleaning of the flats will first be reviewed. Many cleaning recipes have been advocated and a reliable process is the method introduced by Ritschl. The surface is washed with soapy water, and after degreasing say with ether or ethylene trichloride, is rubbed with cotton wool or boiled linen soaked in H_2O_2 (20 vols.). This liquid is a most excellent cleaning medium, removing many organic films and has the advantage too of dissolving away any earlier silver films that may require removal. The final test is the ' breath-figure ' which should be a uniform film, any slight trace of grease breaks up such a film into matt-like droplets. The cleaned glass is then introduced into the vacuum system. A grease film, possibly monomolecular, always remains but this can be removed electrically *in vacuo*.

For the production of aluminium telescope mirrors, Strong recommends deliberate introduction of a controlled grease film of lanoline. The lanoline film is spread over the surface, which is rubbed over with virgin felt. Then precipitated chalk is rubbed over and after further felt rubbing, the residual film removed electrically *in vacuo*. Very tenacious aluminium films are produced in this way. Whatever method is used, all workers are agreed on the final stage, which is the removal of any impurity surface films by passage of an electric glow discharge through the vacuum system before the pressure is too low (say 10^{-2} or 10^{-3} mm. Hg). The ion bombardment removes the surface film. Even here there is variation in practice. Earlier workers preferred the higher voltages of a Rhumkorff coil with small currents, later workers tend to use transformers with volts of some 3000 to 5000 and currents up to 100 milliamps, passing for different times of up to 10 minutes. Strong repeats the procedure several times, flooding the equipment with oxygen each time as this assists removal of film. There is no doubt at all that the absorption of the silver film is profoundly influenced by the success or failure of this impurity film removal, and without ionic cleaning really good films cannot usually be produced.

The cleaning of the various specific surfaces to be coated for study by interferometric methods is a

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Fig. 7.—Tungsten (or molybdenum) strip used as evaporation source in the deposition process. The silver is placed on the loops.

matter requiring judicious consideration. For metals degreasing in a simple thermal condensation degreasing system is all that can be attempted, since most other cleaning agents might produce slight or serious etch. The final cleaning is ionic bombardment preferably for a short time to avoid heat effects. Freshly cleaved surfaces of crystals (*e.g.* mica, calcite, rock-salt etc.) are introduced direct into the evaporation chamber and only mildly cleaned by the glow discharge. Some crystals and plates will tolerate rubbing with hydrogen peroxide to be followed by the usual ionic cleaning.

THE EVAPORATION PROCESS

I Mercury Pumps

The evaporation processes can be conveniently divided into (*a*) the methods of producing the vacuum, (*b*) the means of measuring the pressure, (*c*) the heating method for evaporating the silver, and (*d*) the control of the thickness. I have found that the method of Ritschl has given me the best results and it may be noted that the high quality mirrors described by Kuhn and Wilson were obtained with an arrangement identically the same as that of Ritschl (it so happens that I was a guest research student for a year during 1931 in the laboratory of Paschen in Berlin, and since Ritschl was a member of Paschen's staff and had just then fully developed his technique, I was able to make myself completely familiar with all the details of the method, details which Ritschl was most willing to acquaint me of).

The pump used a four-stage Gaede mercury diffusion pump with an efficient liquid air trap over the throat. The vacuum pot was of drawn brass, completely tinned on the outside. Ports were bolted on back and front and sealed with rubber gaskets. Ritschl strongly advocated internal lining of the pot with an open-ended glass tube, and the ends were roughly closed over with glass discs before placing on the sealing ports.

The pot was horizontal and the surfaces to be coated were placed at each end, some 30 cm. away from a filament at the centre. This was a tungsten spiral and wrapped round it loosely was a fine platinum wire. The silver was hung on wire loops and pre-fused before commencing operations. An unfortunate feature was the ease with which the molten silver drop could fall off as a result of a slight jar. The platinum wire helped by surface tension effects to stop this, but quite an appreciable proportion of silvering operations collapsed through the accident of losing the droplet.

The ionic cleaning was carried out with a Rhumkorff coil giving at least a 3 cm. spark and this same spark was used as a simple yet effective pressure gauge. Attached to a side limb was a small tube with two sealed-in electrodes, distant about 5 cm. When this testing tube showed a hard black vacuum (in the dark) to the spark coil, evaporation was begun. The time to deposit a good mirror was about 2 minutes. The mirror thickness was estimated by viewing a bright lamp through the pair of mirrors formed. I myself became familiar with the colour and intensity characteristics and Ritschl used either the visual judgment (which can be highly critically selective) or else estimated the intensity of the transmitted light with a photometer device.

The most peculiar characteristic of the Ritschl technique is the 'after-treatment.' This is very variable in its effect and deserves a thorough investigation. A mirror when taken from the pot has a deep violet colour (best appreciated by looking through the mirror at the sky). Ritschl found by accident that the gentle waving under it of a glass stopper which had on it a few drops of aqua-regia, the mirror being held face down, led to an extraordinary change in that transparency increased and an image count of say 50 jumped up to perhaps 80. By doing this against a ground glass illuminated screen, one can see the change in colour and transmission spreading over

the surface. The treatment is critical and if over-done the mirror turns brown and is wrecked. The whole change takes a matter of some seconds only.

Finally it was Ritschl's practice to hold the mirror for 1 minute over a crucible containing warmed H_2O_2 (20 vols.). This preserved and hardened the

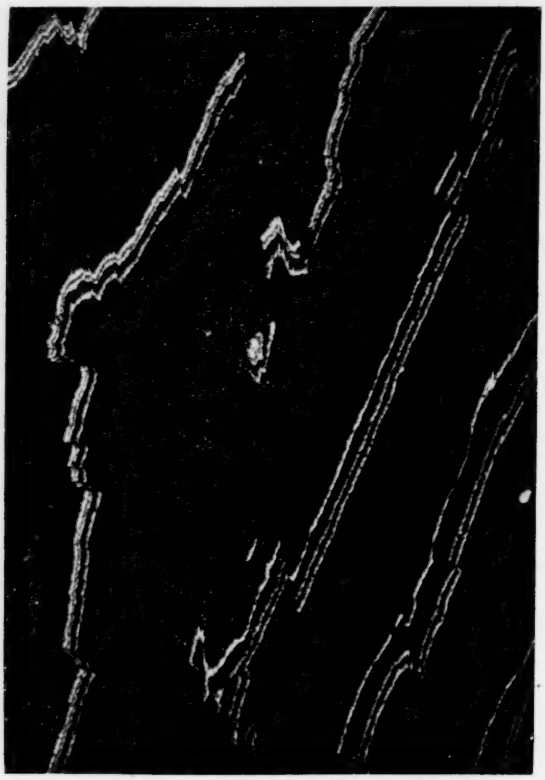

Plate 1.—Multiple-beam wedge fringes given by the face of a diamond (2 sq. mm.).

mirror which would then stand up to scratching with a match-stick.

I have repeated these after-treatments successfully on numerous occasions, but wish to make clear the fact that I have only found this to function with a Ritschl-type of set-up. The aqua-regia effect may act on impurities which come possibly either from the mercury, or from the tungsten or from the platinum, and the matter is certainly worth further investigation. It is the after-treated mirrors that give the exceptionally high counts and the possibility must not be lost sight of that one may not be dealing with simply a silver mirror, but with a complex film, perhaps involving oxide chloride, or even oxy-chloride of silver. Electron diffraction experiments may give the answer.

It is to be noted that a mercury system can be opened to the air with hot pumps for removals or replacement for re-silvering and no complex internal valving systems are required.

There is an indication that the quicker the deposition the better is the film produced.

I have introduced a considerable improvement by the development of a modified filament. This is shown in Fig. 7. A wide strip of tungsten (or molybdenum) is bent into the shape shown. The silver is pushed into the hollow. A current of about 80 amps. is needed to heat the filament. The silver melts and owing to its high surface tension stands up vertically like a molten disc. Thus facing the two receiving surfaces are the large silver areas and only the thin edges of the filament. Any impurities coming out of the filament (*e.g.* tungsten vapour or occluded material) are relatively greatly reduced, since the area of molten silver facing the glass is much more than the area of filament. Furthermore considerable loads of metal can be used without any danger of loss. Multiple loops can be used and indeed different metals can simultaneously be evaporated this way (*e.g.* Ag and Al). I have found absorptions with this filament to be less than those used in the standardised vertical type of plant to be described later when no liquid air traps are used in both instances.

II Oil Pumps

The data given above in (I) were obtained with a Metropolitan Vickers 0.3B pumping set using apiezon oil, and the filament just described. The aqua-regia treatment seems to have no advantageous effect on the mirrors produced this way. The absorptions in (II) are appreciably worse than in (I) and this I attribute to the oil. Liquid air was difficult at the time to obtain and I cannot state whether or not there

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would have been an improvement by introducing a trap (at the expense of pumping speed). Pressure was measured with various gauges (Pirani, ionisation) and approximated to 10^{-5} mm. Hg. Experience shows that the spark test was equally good for deciding on the evaporation pressure. After all the absolute pressure is rarely required. What is needed is some indicator telling when to commence evaporation. The spark suffices. It is advantageous to have a shutter over the receiving surface, to be removed only whilst the evaporation is in progress. With the filament described a large number of mirrors can be made with one loading.

VERTICAL EVAPORATION

The films (III) were obtained with an Edwards vertical type standardised form of 18″ evaporator with a silicone oil high speed pump using a strip electrode carrying the metal, an ionisation pressure gauge for pressure measurement, a mushroom valve to seal the diffusion pump when opening up, an externally controlled shutter to protect the receiving surface, and provision for ionisation cleaning. The advantages of the system are speed, reliability, and ability to handle either many small, or one large surface. It was always used at a pressure lower than 10^{-4} mm. Hg, although the size and layout of gauges and pumps makes it difficult to assess the real pressure near the receiving surface during running conditions. Of course good gauges are an advantage in that they are of some real help in establishing repeat conditions, even if the precise meaning of the pressure recorded during evaporation is obscure. Furthermore their obvious use in tracing possible faults needs no comment. It is re-emphasised that this, as with the other two systems, gives high enough ultimate reflectivities for *thick* films, but for *thin* films the absorption is worse than in the other two cases. This is probably not the fault of the silicone, for I have used a vertical evaporating plant (constructed in the laboratory) using apiezon oil and the absorption is very little different to that of the silicone system. I am inclined to attribute the absorption to material coming off from the hot filament, although I have no real decisive evidence on this point. With a horizontal strip filament in vertical evaporation, the area of hot tungsten facing the receiver far exceeds that of the silver and this I consider to be the cause. In this respect a horizontal evaporator, using the modified filament, offers considerable advantages. Again, assessment of film thickness is more complex in the vertical plant. Either one approximates by viewing the hot filament through the film from above, or one introduces a complex photometric system into the vacuum chamber for estimating the thickness. The former arrangement is a poor one, for the filament brightness itself changes as the mass of silver or it diminishes, and further its colour changes too. Again, one views only through one silver film and this is certainly less sensitive than when viewing through a pair. To some extent one can rely on time of evaporation, but the thickness and absorption are highly critical in the higher reflectivity regions and this always leads to a good deal of uncertainty.

Again, I have never found any improvement in the mirrors from the silicone plant to arise from Ritschl's aqua-regia treatment. On the contrary, it is easy to wreck the mirrors by this violent reagent.

It will be clear from my statements that the question of the dependence of absorption on the nature of the oil used in the pump and on the type of filament is in an uncertain state. This is an aspect well worthy of a serious investigation and it is hoped that the matter will be taken up further by those interested. This is indeed a case where the *art of vacuum technique* rather than the science of vacuum technique is involved, and it is highly desirable that more precise data be obtained on more obscure points.

THE OPTICAL UNIFORMITY OF THE DEPOSIT

The uniformity of both thickness and optical characteristics is a matter of fundamental importance and fortunately there exist some very severe tests of this. Considering first the geometry of deposition from a point source onto a plane disc, the fall off in

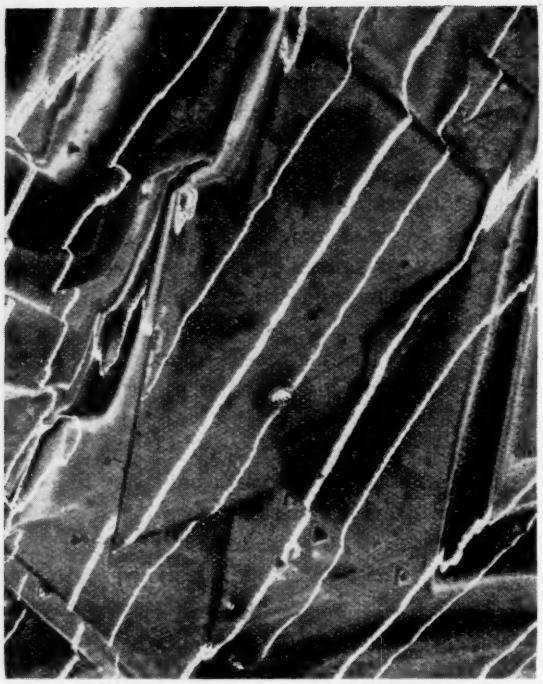

Plate 2.—'Crossed fringes': two superposed fringe systems.

density from the centre to the disc edge is proportional to $\cos^3\emptyset$, if $2\emptyset$ is the angle subtended by the disc at the source. For a disc of radius 5 cm. distant 30 cm. from a *point* source the edge density is 99% that at the centre, on geometrical considerations alone. But in fact the uniformity is much better than this for the source is not a point but extends over an appreciable area, and also inevitable gas collisions and even collisions between metallic vapour atoms in the evaporating beam smooth out the beam. In most work in interferometry a disc of 5 cm. radius more than suffices and in a good deal of the work now being done with the microscope only areas of some square millimetres are required. Here the geometrical uniformity is not involved but rather the question of fine grain uniformity.

I have established that in the macroscopic sense the silver film contours the surface which receives it with remarkable fidelity. Thus there is a considerable reason to believe that numerous crystals cleave in step heights which are simple integral multiples of the unit crystal lattice spacing. These spacings, to take but three examples, have the following values, in Angstrom units.

Mica	Selenite	Calcite
20	15	6

In fact step heights measured on such surfaces coated with silver 500 Å thick have been, for mica, 20, 40, 80 etc. units, with an experimental error of less than 3 Å. For selenite, 15, 30, 45 etc. Å, and for calcite 12 Å (6 Å is perhaps too small to resolve). Not only this, but there is a strong reason for believing that a mica step should remain constant and true to a single 'molecule' along the length of a cleavage line, often for some millimetres or even in special cases, for some centimetres and indeed it has been confirmed that the fringe steps do remain constant. Here then is evidence that deposition of the film closely contours the surface almost to within molecular dimensions, when level changes over macroscopic areas are considered. Particularly striking is the fact that small steps are faithfully contoured even when the silver film is itself 50 times as thick as the step which is under review.

Another piece of evidence is offered by the production of interference filters. I developed these filters in 1942 in conjunction with one of my students and was advised at the time not to publish because of possible war applications. Subsequently these filters were described independently by others. In the filters, silver was deposited onto crude glass (piece of a thin photographic plate). This was followed by a layer of cryolite, and this by a layer of silver. Now it is readily demonstrated that the glass is highly irregular by interferometric standards. Furthermore it can be shown that a change of film thickness of only 3 Å can make a 10% change in the transmitted intensity to monochromatic light or a colour change to white light. Yet despite the very crude surface receiving the deposits large interference filters of very perfect colour uniformity are formed and their band-pass characteristics are surprisingly uniform. Clearly the three successive deposited layers have contoured the crude glass surface almost to within molecular

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dimensions. It is indeed striking that so perfectly uniformly thick a film can be produced on so irregular a glass surface and this is even more striking evidence of the perfection of contouring during evaporation.

Regarding the mechanics of the process, electron microscope studies seem to indicate that the first few atomic layers of silver which come down are rather mobile, almost a two-dimensional gas, and this is responsible for the initial contouring efficiency.

Special problems arise when consideration is applied to the effectiveness of the contouring of small areas. The surfaces of the octahedron (III) planes of diamonds offer an excellent subject here. Such surfaces frequently have on them small triangular pits with sides dropping sharply in some cases. They vary in size and interference fringes can be run across these rectilinear features with a view to establishing the degree of contouring. I have established that the contouring is certainly maintained for elements of area down to a square of side 1/200 millimetre. In any case the optical conditions for multiple-beam interference begins to break down for an element of area of this size. However, there is some distinct evidence that there is slight irregularity at the sharp boundary and if this is borne in mind no errors need arise.

It is of some real interest that when cryolite is deposited over a step edge, a distinct irregularity appears near to the edge. The amount of cryolite on the edge exceeds that in the remaining regions. This might well be a preferential crystallisation on the sharp edge during the crystallisation of the cryolite from the vapour phase. Under such conditions, in making the measurements one must recognise that an irregularity of some hundreds of Angstrom units might locally arise. The effect does not exist in simple deposition of silver alone.

MULTIPLE-LAYER TECHNIQUES

The previous discussion has an important bearing on the question of using the new multiple-layer technique for producing high reflectivity and low absorption. A number of authors, particularly Jacquinot and Dufour, have developed the production of high reflecting films consisting of alternate $\lambda/4$ thick layers of zinc sulphide (refractive index 2.3) and cryolite (refractive index 1.35). Dufour has calculated the following reflectivities for such compound layers.

	R%
1 layer ZnS	31
3 layers ZnS-Cry-ZnS	67
5 layers ZnS-Cry-Zns-Cry-Zns	87
7 layers ZnS-Cry-ZnS-Cry-ZnS-Cry-ZnS ..	95

Cryolite has no effective absorption although ZnS does have some slight absorption depending on the purity. Dufour finds that the 5 layers have an absorption of only about $1\frac{1}{2}\%$ and the 7 layers a little over 2%. The high reflectivity depends on the wavelength, which is a restriction. Dufour, in a personal communication, has informed me that not only does the complex multiple-layer produce high quality multiple-beam fringes, but in addition the final smoothness of an optical surface is distinctly improved, in that local polish marks tend to be obliterated. This is in harmony with my own observations on the edge effect irregularities associated with cryolite deposition.

Whereas this property is helpful in the case of a flat, it is obviously completely undesirable if surface topography is under study. It follows that the *complex multi-layers cannot be used on the surfaces under study*, but can of course be used on the flat, used to match against the surface under study. The preparation of the complex film requires the use of some optical method for evaluating the film thicknesses during the deposition process, without breaking the vacuum. Jacquinot and Dufour have also developed the technique of enhancing the reflectivity of silver films by surface deposition on the silver film of a high-low index pair of dielectric films. The first dielectric film has a thickness a little less than $\lambda/4$ and is the low index film, followed by a $\lambda/4$ high index dielectric film. By this means a silver reflectivity of 86% was raised to 94%, and a reflectivity of 94% to 97% with no increase in absorption, a matter of some real value. Again such a combination can only be used on the flat.

Since these multiple-dielectric or metal-dielectric

Plate 3.—Good wedge fringe quality obtained in reflection using a mercury pumping system.

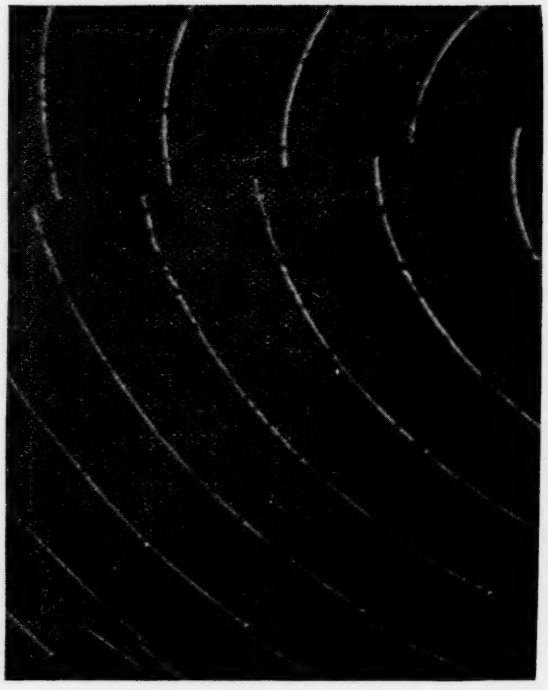
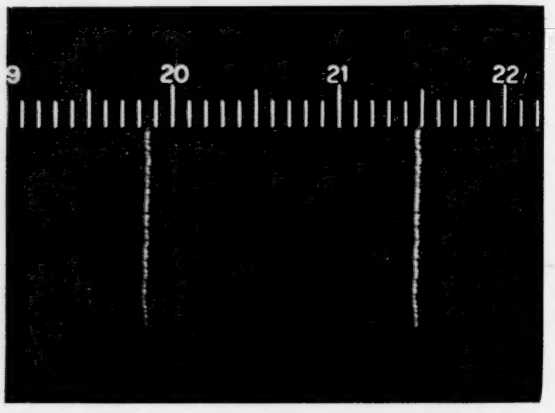


Plate 5.—Cleavage step on mica. Magnification × 100.

Plate 4.—Transmission fringes (white light fringes of equal chromatic order) given by two pieces of polished glass.



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layers are characterised by low absorptions there is considerable future in employing them for the first surface in reflection interferometry.

ILLUSTRATIONS

To conclude this survey a few examples of interferograms will be chosen to illustrate the results obtainable. Plate 1 shows multiple-beam wedge fringes given by the face of a diamond (some 2 square millimetres). The fringes show the mercury green line and the mercury yellow doublet. They are in transmission and both diamond and optical flat were coated in an apiezon oil diffusion vacuum outfit. Attention is drawn to the exceptionally fine definition, showing high reflectivity (some 94%). As the system is in transmission the only effect of absorption is to reduce the intensity.

Plate 2 is a special type of interference picture which I call ' crossed fringes '. Here two fringe systems are superposed. A broad colour tint system which shows up the topography as intensity variations, and on this is superposed a narrow line system like Plate 1. Attention is drawn to the way in which the fringes contour edges and small triangular details, proving that the silver has contoured accordingly.

Plate 3 shows the exceptional wedge fringe quality obtained in *reflection* using a mercury pumping system with trap. This shows a small step on a surface, produced by evaporation of silver onto glass. Not only are the fringes extremely sharp, but at the same time the contrast is very high, proving that the absorption on the front film is quite low.

Plate 4 shows fringes in transmission, white light fringes of equal chromatic order, given by two pieces of polished plate glass. Each fringe is the line profile for a one millimetre length on the surface. It will be seen that scratch polish marks about 1/100 mm. wide are revealed, indicating the contouring ability in extension across the surface.

Finally Plate 5 shows a cleavage step on mica (magnification is × 100) and the sharp edge discontinuity is clearly rendered, again further evidence of satisfactory contouring.

In all these pictures, the first silver film (both films in the transmission fringes) is about 500 Å thick, *i.e.* a thickness corresponding to 1/5th of an order separation, yet details of 1/250th of an order separation are measurable under the microscope.

It is hoped that this survey will have demonstrated the basic importance of a correct vacuum technique for multiple-beam interferometry. One can add that high-quality reflecting films alone will not lead to fringes of the quality shown here. The associated optical conditions are quite critical and only if these conditions are satisfied can the good mirrors be made to yield narrow high contrast fringes.

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TRAINING IN VACUUM TECHNOLOGY

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Summary

THE author discusses the subject of training for vacuum work and suggests that there is a need for special courses for post-graduates or technicians who have completed their basic training. The Northampton Polytechnic, with which the author is associated, has arranged for a course on vacuum technique as a permanent feature of its educational programme and the author gives a detailed description of the syllabus of the course, the laboratory facilities available and the practical work included in the course.

Sommaire

L'AUTEUR DISCUTE le sujet de la formation du personnel chargé des travaux de vide et suggère des cours spéciaux pour Ingénieurs et Techniciens ayant terminé leurs autres études. A l'Ecole Polytechnique de Northampton, (Londres) à laquelle l'auteur est attaché, a été établi un course de technique du vide faisant partie permanente de son programme d'étude. L'auteur donne un apercu du plan d'étude, des laboratoires et travaux pratiques faisant partie du cours.

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THE PURPOSE of this article, written at the request of the Editor of 'Vacuum', is to raise the general question of training in Vacuum Technology and to describe by way of illustration the provision made for it at one particular College of Technology, the Northampton Polytechnic, London.

GENERAL

In the last decade or so vacuum technique has developed so vigorously that it has now emerged from the status of a laboratory art, depending on individual skill and experience, and become a branch of engineering. This transformation has brought with it the demand for a considerable number of workers at various levels with special knowledge and experience in this field. The people required may be divided roughly into three classes :

I Physicists or engineers, to work on the design and production of vacuum equipment.

II Scientists or engineers to be occupied with the applications of vacuum technique both in research and manufacture.

III Skilled technicians, who may be engaged in the day to day maintenance of vacuum plant or as assistants to either of the above.

Until recently members of each of these groups have picked up their specialist knowledge as best they could in the course of their job from their colleagues or by reading and in many cases this has produced excellent results. But under present-day conditions with the growing complexity and range of the subject there seems no doubt that there is a strong case for the provision of some specialist instruction in vacuum technique. In some cases a short course could be appropriately included at the undergraduate level and some colleges do this already in their curriculum for the Electrical Engineering or Physics degree, or Higher National Certificate in Applied Physics. But, at most, this can provide only an introduction to the subject and is neither adequate nor intended to fit a man for responsible work in Vacuum Engineering. For this purpose special courses are needed which can be taken after degree or other basic training ; and the question arises as to the best form in which such courses can be provided. One solution is for them to be arranged as evening or part-time day classes. Despite the well-known difficulties of part-time education this is sometimes a satisfactory

* MS. Received April, 1951.

arrangement. It does however depend upon the provision of adequate laboratory facilities within easy travelling distance of the student's place of employment. While it is likely that many colleges will be able to provide sufficient equipment for an introductory course, it is improbable that fully equipped High Vacuum laboratories will be available in more than a few centres. It appears therefore that there is much to be said for the organisation of short intensive full-time courses. Apart from considerations of geographical convenience such short courses have some obvious advantages over the longer drawn-out ones lasting over a session of some 30 weeks, particularly so perhaps for the graduate type of student who is used to making a concentrated effort of learning. It is felt that sufficient ground could be covered in a period of about 4 weeks to make it profitable for a firm to send a man on such a course. For example, in the case of a newly-appointed graduate the appropriate time would be immediately after his appointment. On the other hand for a technician it is probable that the most suitable time would be after he had completed some basic training and was due to undertake more responsible duties.

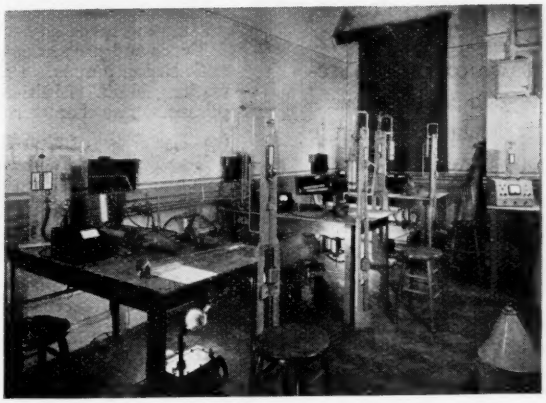

Fig. 1.—View of laboratory showing three pumping stations.

A six month post-graduate course in Electronics and High Vacuum Technique was conducted on two occasions—at the end of and shortly after the war—by the Electrical Engineering Department of Manchester University. The students were Engineering and Physics graduates from the staffs of valve manufacturing firms for whom the course was specially arranged. Vacuum technique formed a considerable section of the course and a special laboratory—almost certainly the first of its kind—was set up to deal with the practical work. This laboratory is at present used in connection with courses on Vacuum Technique forming part of the undergraduate training for Engineering and Physics students, and also for research purposes.

The rest of this article gives an account of the courses and laboratory facilities provided at the Northampton Polytechnic, in London.

REVIEW OF NORTHAMPTON POLYTECHNIC VACUUM COURSES

For some years before the war a certain amount of introductory work on Vacuum Technique was included in an Applied Physics course which formed part of the curriculum for full-time Engineering Degree students. The practical work included in this was very limited in scope because of lack of space and equipment. After the war it was realised that there was need for far more adequate provision for the teaching of the subject and, with the encouragement of a number of people in industry, it was decided in 1945 to set up a High Vacuum laboratory. It was not until the end of 1948 that the necessary space was available ; since then considerable progress has been made in developing suitable laboratory experiments.

Since the war the policy of including a short course of lectures on Vacuum Technology for Electrical Engineering Degree students has been continued. The recent introduction of Electronics as a separate subject in Part II of the London University Engineering Degree examination has led to some extension of this course, particularly on the experimental side. In addition to the work on vacuum technique the laboratory has been used for experiments on gas-

discharge phenomena. Vacuum technology also forms a section of the work in applied physics contained in the Higher National Certificate Course in Applied Physics at this Polytechnic. This whole course extends over two years ; some students attend for three evenings and others one full day per week.

The Polytechnic has also conducted every year since the war a special evening course in Vacuum Technology. For the first three sessions this consisted of lectures only, occupying one evening per week throughout the session, *i.e.* approximately 30 weeks. Since 1948, however, when the new laboratory was brought into operation the general plan has been to devote two evenings per week to the course, one for lectures and the other for practical work. The lecture course is given in two concurrent sections ; one for students with a good scientific background of approximately degree standard and the other for those with a more modest training. These are appropriate, roughly speaking, to scientific staff and technicians respectively. The first twenty or so of the lecture periods—each of about 2 hours—are devoted to the fundamentals of the subject and the remainder to a series of lectures on industrial applications. The latter are given by specialists from industry and their generous and very valuable co-operation in this way is very much appreciated.

The number of students attending these courses has varied from year to year with an average of over twenty. They have included people from each of the three groups mentioned earlier in this article ; for example :—graduate scientists (physicists, engineers and chemists) from government and industrial research laboratories, and from lamp, valve, chemical and vacuum engineering firms ; post-graduate university research students ; and technicians and technical administrative staff from the electrical and chemical industry.

LECTURE SYLLABUS

The following syllabus forms the basis of the special evening course. The actual treatment naturally depends on the standard of the particular group of students concerned. For example, the kinetic theory is treated descriptively for the less advanced students but with more mathematical elaboration for those of degree standard.

Syllabus

(1) Kinetic Theory of Gases

Basic concepts of the theory. Pressure and temperature. The distribution of velocities. Mean free path. Viscosity and thermal conductivity of a gas. Effusion. Thermal transpiration.

(2) The Measurement of Low Pressure

Units of pressure.

The theory and properties of the McLeod gauge, Pirani hot-wire gauge, thermo-couple gauges, the hot and cold-cathode ionisation gauges, the radioactive ionisation gauge, the Knudsen gauge, etc.

The use of gauges.

(3) The Production of High Vacua

Pumping speed and throughput.

Types and properties of backing pumps.

Diffusion pumps. Principle of operation. Choice of working fluid. Multi-stage, fractionating and booster pumps. The characteristics and use of diffusion pumps.

(4) The Flow of Gas Through Tubes

The Poisseuille and Knudsen regimes. The effect of connecting tubing on the effective pumping speed and on the measurement of pressure. Design of vacuum systems.

(5) The Measurement of Pumping Speed

(6) Vacuum Plumbing

Choice of materials. Demountable systems, method of sealing, vacuum valves, methods of producing motion *in vacuo*, etc.

(7) Leak Finding

Discussion of available methods.

(8) Gas-Solid Phenomena

The adsorption and solution of gases by solids. Degassing and gettering.

(9) Glass-to-Metal Seals

Types and properties of glass. Annealing. Strain measurement. Types of glass-to-metal seals and methods of production.

(10) Industrial Applications of High Vacuum Technology

This series of special lectures dealt with the following topics :—

Valve Production.

1. Materials and their pre-treatment.
2. The pumping of transmitting valves and cathode-ray tubes.
3. Receiving valve production.

Optical Industry.

1. General technique of evaporation and sputtering.
2. Metallising and blooming of glass surfaces. Production of interference filters.

Vacuum Metallurgy.

Melting, casting and sintering *in vacuo*. Refining metals *in vacuo* and the preparation of metals by dissociation of oxides.

Dehydration and distillation *in vacuo*. Freeze drying. Industrial applications of vacuum coating techniques.

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LABORATORY FACILITIES

The laboratory has an area of about 650 sq. ft. and in addition a small room opening off the laboratory has been fitted up as a local workshop. This latter is equipped with a lathe, drilling machine, grinder, welding plant, etc., and it is proving of the greatest value in the development and running of the laboratory particularly as a great deal of the apparatus has necessarily had to be made on the spot. Some additional storage space for equipment is provided in a third room near by.

The laboratory contains five pumping stations, each equipped to enable students to carry out a wide variety of experiments on the fundamental principles of vacuum technique. In designing these stations the main considerations which had to be borne in mind were the following :—

(*a*) It is desirable to avoid on the one hand presenting the student with a completed set-up in which he has only to take readings, for he then learns little about vacuum technique.

On the other hand, it is impracticable for him to construct the apparatus for each experiment from scratch in the time available which is, at the most, about 3 hours. Furthermore it is not in general possible for the system to be left assembled for several weeks as the stations are required by students in other courses. Thus demountable equipment is used wherever possible.

(*b*) It is desirable that the students should use equipment that resembles that which they are likely to meet in practice. The stations are, therefore, equipped with as wide a variety as possible of modern types of pumps, gauges, etc. Metal diffusion pumps are used as standard equipment ; these are mainly oil diffusion pumps although some mercury pumps are available. A good deal of metal is used in the construction of the systems.

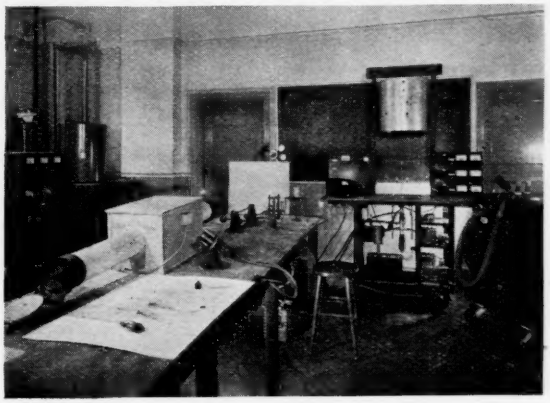

Fig. 2.—View of laboratory showing evaporating plant and valve pumping station.

Some of the pumping stations are shown in the photograph (Fig. 1). Each consists of a bench 5 ft. x 3 ft., the top being made of ' Sindanyo ' board so that it can be drilled and cut away as required. The backing pump is on the floor and the diffusion pump is mounted below the bench level, being supported on 3 vertical rods which are attached to the under-side of the table so that it can readily be adjusted in height or replaced by another type. The backing pump is connected to the system via an oil reservoir, 3-way tap and glass P_2O_5-trap ; these are, however, now being replaced by metal traps fitted with an air release valve and diaphragm outlet valves.

A ' header ' is mounted on the diffusion pump just above bench level (see Fig. 1). The purpose of this is to act as a distribution board so that the apparatus required for any experiment can be readily connected to the basic pumping equipment. The header consists of a cylindrical metal tube $2\frac{1}{2}''$ in diameter, with a T-piece of a diameter of $1\frac{1}{4}''$ leading from it. The ends of the cylinder and of the T-piece can be vacuum-sealed by O-ring seals. Other outlets are provided for gauge connections etc. by two sizes of metal cone joints which have been constructed to match the standard glass cone joints available commercially. These cone joints have been so placed that whatever the position of the header, one cone joint is vertical ; thus a heavy Philips gauge can be inserted without the need for clamping. The header can be constructed of steel or brass ; if the latter, it must be heavily nickel-plated as mercury may be present in the system. It has been found that this

device has greatly increased the flexibility of the pumping stations and very much simplified the running of the laboratory.

Each pumping station has a McLeod gauge and liquid-air trap permanently mounted at the end of the bench. Various other types of gauges are available as required.

The supplies laid on to each station consist of water, gas, compressed air, and electricity ; in the case of the latter, the supply terminals at each bench are brought to a distribution board containing one 15-amp. and six 5-amp. sockets. The five pumping stations are arranged round the walls to simplify the supply problem. A bench running down the centre of the laboratory is equipped for glass-working using either air-coal gas or oxy-coal gas. Mercury and water stills have been installed and a mobile G.E.C. eddy-current heater is available.

The laboratory is provided with an evaporating and sputtering unit (Fig. 2) with 18″ bell jar and high speed pumping equipment. Also we have recently constructed equipment for the manufacture and processing of small valves (Fig. 2). This includes hydrogen and vacuum furnaces, spot-welder, a small sealing-in machine, and a valve pumping station. The latter is a mobile unit built up on a trolley base and is complete with pumps, baking oven supported on a gantry, electrical power supplies and measuring instruments.

EXPERIMENTAL WORK

The following list gives an idea of the kind of experiment which has so far been developed :—

1. Dismantling and examination of backing pumps, diffusion pumps and other items of high vacuum equipment.
2. Absolute calibration of a McLeod Gauge.
3. Measurement of pumping speeds of 1- and 2-stage backing pumps. Speed measurements are carried out by admitting the gas through a needle valve from a speed-measuring pipette.
4. Experiment on methods of leak location.
5. Calibration of the Pirani gauge for various gases. The effect of water vapour on McLeod and Pirani gauges is also investigated.
6. Calibration of the ionisation gauge for various gases.
7. Investigation of the performance of a diffusion pump at various fine-side pressures, backing pressures and heater power. The speed is also measured for hydrogen.
8. Investigation of flow through tubes. The effect of connecting tubes on the effective speed of a pump.
9. Investigation of the time required to pump down two identical vessels, one of glass and the other of metal. The effect of a preliminary removal of water vapour by exposure to P_2O_5 is also investigated.
10. *Degassing*. A glass tube containing metal and a getter is subjected to the processes used in obtaining a high vacuum in a sealed-off system. The apparatus is designed so that the pressure changes can be followed thus allowing the effectiveness of the various processes to be assessed. The experiment is a valuable introduction to the technique of valve manufacture.
11. *Valve Construction*. In this experiment the students construct a small valve. This involves pre-treating the electrode components, assembling them on a pinch, sealing-on the bulb, pumping and processing the valve and mounting it on a base. They then age and test it.
12. *Experiments on Evaporation and Blooming*. The students produce an aluminised mirror, bloomed lens and an interference filter.

CONCLUSION

It is hoped that the above account of what is being done at one place in this new field of education and training in Vacuum Technology may be of interest, and possibly of some assistance to other places where similar developments are being planned, and that it will be followed in the pages of this Journal by further contributions on the subject both from Colleges and Industrial people concerned with Vacuum Engineering.

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INVESTIGATIONS INTO THE ULTIMATE PRESSURES of diffusion pumps designed to purify their working fluids

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Summary

A DIFFUSION pump which powerfully purifies its fluid from contaminants and decomposition products can produce very low pressures as quickly with a fluid of commercial quality as it can with a specially prepared fluid. This is demonstrated by comparative tests on pumps designed to purify continuously their working fluids with varying efficiency, and operated with proprietory pumping fluids and with commercial plasticisers.

Fluid decomposition may contribute both permanent gas and vapour components to the ultimate pressure so that in certain cases only a moderate improvement in 'ultimate' can be obtained by the use of a refrigerated trap. In a pump designed to keep the concentration of decomposition products in its fluid at a very low level, the rate of evolution of gaseous products is reduced, probably because intermediate products of fluid breakdown tend to be removed before they finally decompose into gases. In addition to the much lower total pressure which may be obtained by such a pump, the application of a refrigerated trap may be much more effective.

These advantages can be obtained in a pump of rugged and simple design without prejudice to other performance characteristics.

Sommaire

UNE POMPE A DIFFUSION qui puissamment purifie son fluide de produits contaminants et décomposants peut produire de très basses pressions aussi rapidement avec un fluide de qualité commerciale qu'avec un fluide préparé spécialement. Ceci est démontré par des essais comparatifs avec des pompes construites de telle sorte qu'elles purifient continuellement leur fluide de fonctionnement, mises en travail avec des fluides spéciaux et avec des plastifiants du commerce. La décomposition du fluide peut donner naissance à des gaz parfaits et à des vapeurs qui influencent le vide limite, de sorte que dans certains cas l'utilisation d'un piège réfrigéré n'apporte qu'une médiocre amélioration de la limite. Dans une pompe spécialement construite en vue de maintenir la concentration de produits de décomposition dans son fluide a un niveau très bas, le régime d'évolution de produits gazeux est réduit, probablement par suite de l'évacuation des produits intermédiaires de la décomposition du fluide avant leur transformation finale en gaz. En tenant compte que l'on peut obtenir avec une telle pompe une pression plus basse, l'application d'un piège réfrigéré est probablement bien plus efficace. Ces avantages peuvant être réalisés dans une pompe de construction simple et robuste, sans préjudice pour les autres performances.

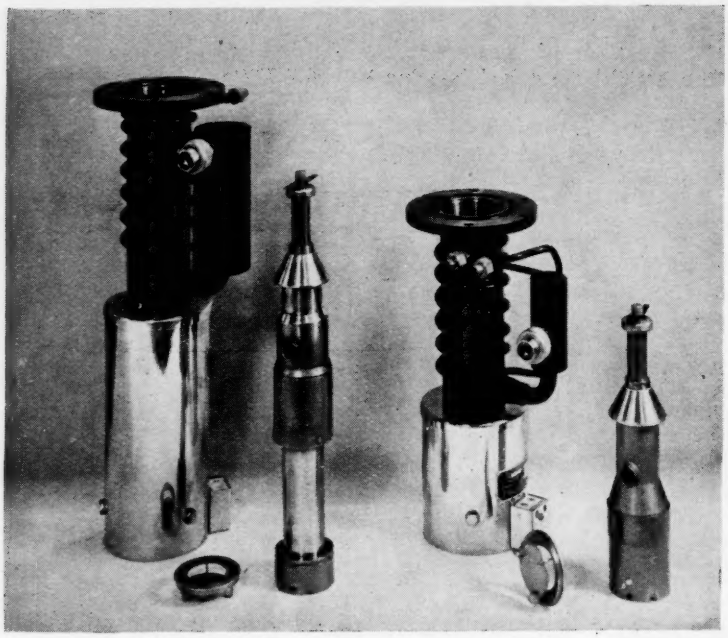
PREFACE

THE DESIGN of an oil diffusion pump for general use usually involves considerable compromise. The need for ruggedness dictates a metal construction, and ease of manufacture and of installation are advocates of the conventional vertical tubular body with a closed lower end to form the boiler, and containing umbrella stages.

A high pumping speed, a substantial limiting backing pressure and the consistent ability to produce a low ultimate pressure are the desirable performance characteristics, and the first two are readily obtainable with the construction described by using a multi-stage interior and sufficient heater input. A consistently low ultimate is, however, less easy to achieve with this construction.

Both Hickman [1] and Jaeckel [2] have shown how seriously light vapours and gases, produced by thermal decomposition of the fluid, can affect the ultimate, and have emphasized how greatly decomposition is accelerated by a metal construction and a high boiler temperature. Hickman [1] has pointed out how readily

* M.S. Received March, 1951.



View of pump B (on right) and pump C. Pump A is identical with pump B except for an extra cooling turn (the interiors and baffles are shown beside the respective pump bodies).

the vertical pump with umbrella stages distils light fractions of operating fluid towards its high vacuum end, and how inefficient conventional fractionation is likely to be in a small and comparatively deep boiler (this sort of boiler is inherent in the vertical design when it is applied to the small bore pumps which are used in so many very low pressure applications). Both workers have also pointed to a partial solution through the distillation of volatiles completely out of the pump and into the backing line. To this end Jaeckel has used the cumbersome arrangement of a backing diffusion pump and Hickman, who avoids the simple construction described, has directed the last stage of some of his pumps upwards into an alembic system, and has used a similar principal in horizontal metal pumps.

In his most recent paper [4], Hickman discloses how even the most favourably designed fractionating pump is a much less effective fractionator than was supposed, and the complete ejection of volatiles from the pump is shown to be correspondingly more desirable. This interesting contribution, which came to our notice when our work was nearly completed, served to endorse our own views on pump design, for in the small pumps to be described we have abandoned any attempt at conventional fractionation (in the sense that different constituents of a fluid are encouraged to accumulate in different boilers or compartments of a boiler) and have concentrated chiefly on the efficient ejection of undesirables entirely from the pump.

It is evident from the literature that there is considerable disagreement about the lowest pressure that can be achieved by a diffusion pump using a particular fluid. Hickman, for example, finds that with Octoil-S a pressure of 5×10^{-8} mm. Hg can be obtained [1,3,4]. Blears [5], using the same fluid in a different pump obtained only 5×10^{-6} mm. Hg. Both workers were using ionization gauges for pressure measurement.

These three factors may influence the results with a given fluid :—

1. Pump characteristics.
2. Variations in the quality of the fluid.
3. Method used for measuring the ultimate pressure.

This paper is concerned with the first and second of these factors. With regard to the third, many workers have shown how easy it is, by the use of different gauging techniques, to obtain completely contradictory results with the ionization gauge, and Blears, in particular, has shown that some of the differences found are due to tubulation errors in the gauges. Whereas Hickman appears generally to use tubulated gauges, Blears used his ' high speed ' type to make his measurements and claimed that this gauge normally indicates a pressure from three to a hundred times higher than does a tubulated gauge connected

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to the same header.

The authors have used Blears' arrangement of 'high speed' gauge throughout—a bare electrode assembly freely suspended in the test header. To what extent their results may be compared with those of other investigators using tubulated ionization gauges is unknown.

INTRODUCTION

The *Atmosphere* in a chamber being pumped by a diffusion pump may comprise :—

(a) Gases or vapours supplied from leakage or from outgassing of chamber walls.

(b) Gases or vapours which have returned from the backing side of the pump by back-diffusion through the vapour jets, or by solution at backing pressure in the condensed pumping fluid returning to the boiler followed by liberation from the boiler through the jets.

(c) Gases and light vapours produced by the continuous decomposition in the boiler of some of the pumping fluid, and afterwards liberated through the jets.

(d) The vapour of the pumping fluid itself or of its constituents (some of which may be impurities of comparatively high vapour pressure).

The contribution of chamber leakage and outgassing to the ultimate attainable pressure can be reduced by high pumping speed, but it is best controlled by good chamber design and construction.

The return of gases, etc. to the fine side by back diffusion can be made negligibly small without difficulty in a multi-stage pump, by making the jets sufficiently dense.

We are more concerned at present to consider designs of pump which limit the passage to the boiler of gas or vapour dissolved in returning pump fluid, designs which rapidly purge themselves of high vapour pressure liquids present as initial impurities in the pumping fluid or produced by its decomposition in the pump, and designs which direct vapour from the lighter constituents of a mixed fluid preferentially to the lower stages of the pump so that their vapour pressure makes a reduced contribution to the ultimate. Though not all the design features to be described are new, some of them do not seem previously to have been fully exploited.

SELF-PURIFYING FEATURES OF THE PUMPS USED

(1) A Purifying Arrangement for Gases and Light Vapours

The returning condensed pump fluid normally flows down to the boiler of a conventional pump through a region exposed to the backing pressure. It has been argued that gases can dissolve in the returning fluid at this pressure and light vapours can condense and mix with it. These undesirables are then carried down into the boiler, where they are released to emerge from the jets and possibly to make a contribution to the ultimate pressure in the chamber. The higher the backing pressure, the greater the quantity of dissolved gas and the greater the effect on the ultimate is likely to be.

Some workers have arranged diffusion pumps in series, one to back the other. The low backing pressure provided by the second pump assists in stripping off dissolved gases and undesirables from the fluid return of the first, and reduces any chance of fluid oxidation.

The three-stage pump illustrated in Fig. 1 (Pump A) has the third stage jet working in a side tube leading to the backing spout. This jet is capable of maintaining a low pressure (below 10^{-2} mm. Hg) in the region Z independently of normal variations in backing pressure. All the condensed working fluid including that from the side jet returns to the boiler down the pump walls in region Z and is spread as a thin film over these walls. The pump walls in this region are allowed to run as hot as is compatible with proper return of the working fluid to the boiler. These conditions encourage the rapid stripping of dissolved gases and volatiles from the returning fluid, or indeed may prevent their condensation or solution. The undesirables are pumped by the side

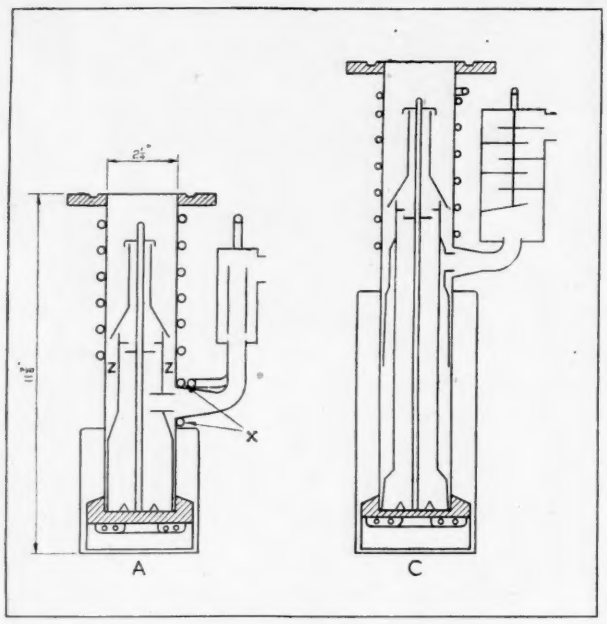

Fig. 1.—Diagrammatic representation of pumps A and C. Pump B is identical with pump A, save that the cooling turn 'X' is omitted.

stage and are thrown out into the backing line.

(2) A Purifying Arrangement for Impurities of Lower Vapour Pressure

Liquids having vapour pressures somewhat higher than that of the pump fluid may be present in it as initial impurities or may result from the thermal decomposition of the fluid in the boiler. These impurities may not be sufficiently volatile to be dealt with completely in the manner described above, but at least a large part of them can be effectively eliminated from the pump by introducing a slight variation to the design of Pump A.

The cooling turn (marked X in Fig. 1) round the conical brass entry to the side tube is removed, and the side tube is allowed to run hot—being heated by the impinging jet. Cooling is maintained on the backing condenser which contains a small trap for liquid. Thus modified the pump is designated Pump B.

The side tube now appears to act as a discriminating condenser which condenses only the heavier fractions of the vapour supplied from the jet. The slightly lighter fractions condense less readily and tend to pass into the backing condenser and indeed right out of the pump in the vapour state. Only part of the vapour produced in the boiler goes to feed the side jet, but all must in time take its turn through the jet and elimination of undesirables is continuous. It was thought at first that the pump with the uncooled side tube might lose appreciable quantities of its fluid charge but in fact there is no such loss. The undesirable lighter fractions constitute, of course, a minute quantity of liquid.

(3) An Arrangement to Direct Volatile Impurities (Preferentially) to the Side Stage.

Pump C in Fig. 1 has a similar arrangement of stages to the ones previously described. In this case, however, the condensed fluid returning to the boiler passes a skirt below the stages and then falls as a thin film down the elongated lower portion of the pump wall before entering the main boiler. This elongated portion of wall is heated from below and is surrounded by a radiation shield. It forms an outer boiler from which vapour is supplied to the side stage only. Lighter fractions of fluid tend to vapourize first and the thin film flow makes it easier for them to do so. The liquid which finally enters the main boiler is to some extent purged of light fractions ; the top two stages are fed from this boiler, so the vapour pressure of operating fluid above the top stage should be correspondingly reduced.

This arrangement directs light fractions of fluid preferentially to a stage which has been designed to eliminate undesirably light components entirely from the pump. The arrangement combines a degree of fractionation with a more specific self-purging action than was possessed by the last pump described.

Water cooling was omitted from the backing condenser of this pump, which was made larger and of somewhat different design.

Temperature measurements of the hot wall under the radiation shield and of the boiler base are plotted in Fig. 2 for various working fluids and heater inputs.

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Thermometer pockets were brazed to the wall for these tests, and the base was drilled to take the thermometer.

The ways in which we consider the various arrangements work have, for simplicity, been described dogmatically. These were the arguments we used when the pumps were evolved and the experimental results to be presented later will provide some evidence to help estimate their accuracy.*

PUMPING FLUIDS USED

A good pumping fluid combines a sufficiently high molecular weight with a very low vapour pressure at room temperature. It is free from impurities and it is chemically stable at pump boiler temperature. Ideally it will survive the repeated admission of atmospheric air to the pump while hot. Other desirable qualities do not concern us here.

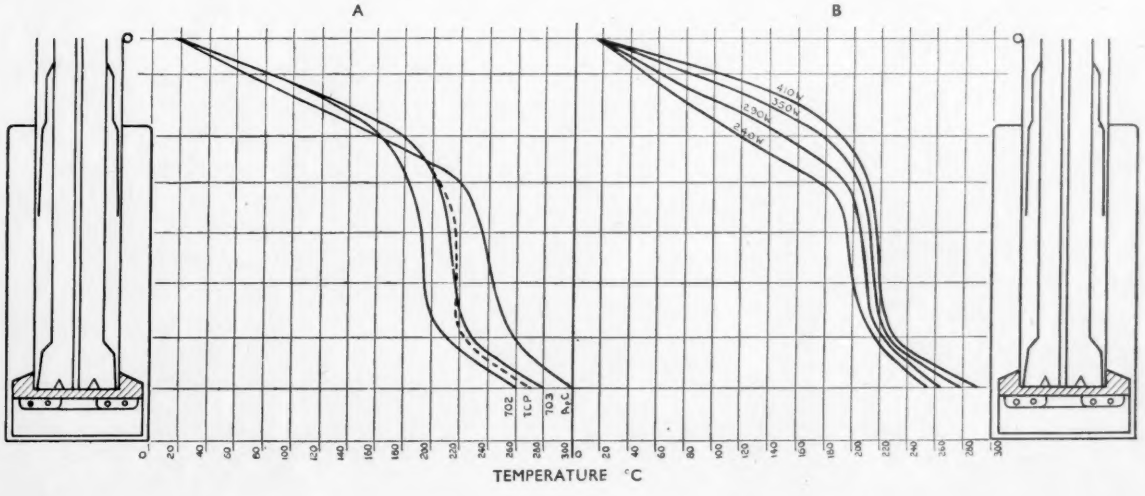
Great purity is obtained by discreet selection of materials and meticulous care during preparation[4], prepared pumping fluids are therefore expensive.

Chemical stability is an inherent property of the fluid selected. Rate of breakdown is increased by high boiler temperature or by the presence in the pump of materials which act as catalysts. Sometimes the breakdown is autocatalytic so that decomposition is continuously accelerated by the accumulating decomposition products. Permanent gases may be the final products of a breakdown in several stages.

The influence of thermal breakdown on the lowest pressure obtainable is a major topic in this paper. It is perhaps appropriate to point out here that poor chemical stability in the pumping fluid can lead to other troubles which form no part of our present subject. In routine use, where the pump is handling considerable quantities of air, or where operational mistakes may occur, most pumping fluids tend to decompose more or less severely. The pump may be capable of eliminating volatile decomposition products and its performance may remain satisfactory for a considerable period, but heavy deposits of carbon may accumulate on the jets and in the boiler. In a severe case, pumping eventually ceases altogether, and the pump is found to be empty of fluid and choked

* The pumps used for these investigations and referred to as A, B and C were an early version of the Edwards 203 and the present 203 and F203 types respectively. Standard heaters, " O " ring gaskets and insert baffles were used. Patents are pending on some features of the designs.

Fig. 2.—Curves of body-wall temperature below cooling turns for pump C. (*a*) With various fluids at 350 watts input, (*b*) with Silicone 703 at various inputs.



with carbon which is difficult to remove. It is the particular virtue of Silicone fluids that they are immeasurably superior in this respect to all other pump fluids except mercury. We hope to deal more fully with this aspect of fluid stability in a later paper.

Turning again to the influence of fluid purity and chemical stability on the ultimate fine pressure, a pump having a powerful self-purging action should be able to work with a fluid of ordinary commercial quality and to purify it in a reasonable time so that the ultimate obtained compares with that obtained using a highly refined product. Such a pump should be able to work with a fluid subject to some decomposition at the boiler pressure desired, and should eliminate the decomposition products so rapidly that a useful ultimate can be obtained. If the decomposition is autocatalytic, then the rapid removal of the products will actually reduce the decomposition rate. In so far as permanent gases are final products of a complex decomposition process, the rapid removal of intermediate products may have a profound effect on the rate of gas evolution.

A fluid that is a discreet chemical substance seems more likely to be readily ' conditionable ' than does a fluid composed of a mixture of related substances, for in the first case the impurities to be eliminated and the fluid itself are likely to have greatly different vapour pressures, and the pump can more easily discriminate between them.

The following fluids were selected for test. The plasticisers were all discreet chemical substances. The abbreviations in brackets have been used when presenting results.

Prepared Fluids	Plasticisers
Silicone 702 (702)	M. Tri-Cresyl Phosphate (TCP)
Silicone 703 (703)	Tri-Xylenyl Phosphate (TXP)
Apiezon B (Ap. B)	Di-Ethyl Hexyl Sebacate (EHS)
Apiezon C (Ap. C)	

It is hoped later to extend both these lists.

EXPERIMENTAL APPARATUS

The apparatus is illustrated in Fig. 3. The three diffusion pumps already described are connected to a common backing manifold. A ' Vacustat ' McLeod gauge, a Pirani gauge, a drying trap, a fine control needle valve and an air admittance valve are also connected to the backing manifold. This is pumped by a rotary backing pump through a small diffusion pump which is not normally in use.

Each diffusion pump has a standard fixed baffle in its mouth and a metal header mounted above it on a standard 'O'-ring rubber gasket. The headers carry ionization gauge electrode assemblies freely supported inside them. They are wound with copper pipe outside for water cooling and have troughs for stronger cooling with solid CO_2.

Each pump and header has a separate water supply from a common main. The water normally flows first down through the header and then down through the pump, the two being in series. There are arrangements to by-pass the header

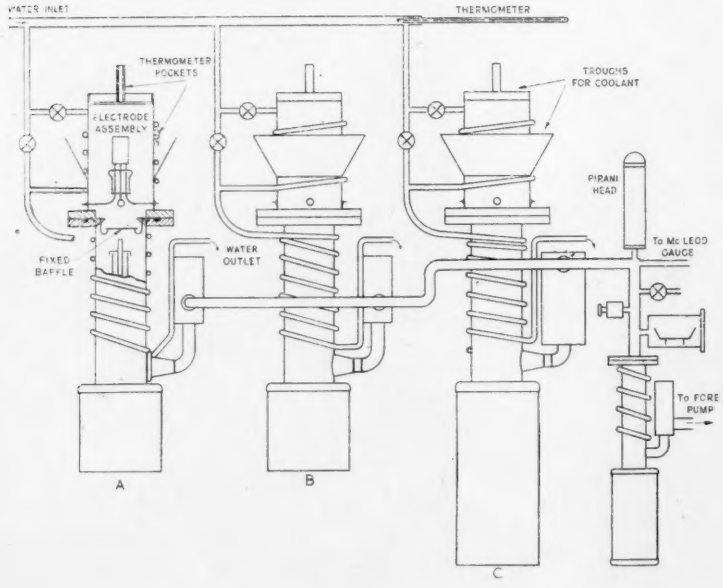


Fig. 3.—Diagrammatic representation of the experimental apparatus.

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if desired.

An alternative water supply can be provided for any one pump and header when desired. This consists of a mains-fed constant head apparatus and a water heater controlled by a 'Variac' and permits the supply of warm water at controlled temperature.

Thermometers are suitably disposed in the water lines and on the headers.

The pump A has a water cooling turn round the side tube near the pump body. Arrangements were made to by-pass this cooling turn when desired. Pump A becomes identical with Pump B when this is done, save for a difference in heater input.

The standard heater inputs are 350 watts for the Pumps A and C and 250 watts for the Pump B. A 'Variac' voltage regulator allows these inputs to be varied when desired.

EXPERIMENTAL METHOD

(1) Preparation of Apparatus

Before each test run with a particular fluid, the three pumps and headers were dismantled, and all parts washed in organic solvent. The pumps were reassembled, mounted, and charged each with 50 ml. of the fluid to be tested. The electrode assemblies were fitted with new tungsten filaments, remounted in the headers and calibrated for air (see Appendix). The headers were re-washed to remove any contamination of fluid from the calibration pump, and were baked for about two hours in a vacuum oven at less than one micron pressure and at about 150°C. temperature. The baffles from the pump mouths were baked at the same time. The parts were taken from the oven and immediately mounted on the diffusion pumps, and the apparatus was at once evacuated by the rotary pump.

(2) Test Runs

A test run with one fluid usually lasted about two weeks. During a complete run, the tests listed below were made in the order indicated. During some of the runs one or two tests were omitted, where it was thought they would yield little new information.

(1) Pressure-time curves were taken during the initial pump-down.
(2) Observations of fine pressure were made with various header temperatures.
(3) Fine pressures were observed after the headers had been strongly cooled.
(4) Fine pressure above Pump A was observed after the flow of cooling water to the side stage had been interrupted and after the heater input had been reduced.
(5) Observations of fine pressure were made with various heater inputs.
(6) Observations of fine pressure were made with various backing pressures.
(7) Pressure-time curves were taken during pump-down after the hot pumping fluid had been exposed to atmospheric pressure.

After each run the three pumps were dismantled, the pump interiors and the fluid charges were examined, and the ionization gauges were re-calibrated to check for variations during use.

(3) Making the Observations

No special outgassing techniques were used on the gauges. After each change in fine pressure sufficient time was allowed for the gauge indications to become sensibly steady before the readings were taken. Cooling water temperature and backing pressure were noted whenever a fine pressure was recorded.

After each test involving a departure from normal pumping conditions (as indicated in the description of the apparatus), the normal conditions were restored and the pumps were given time to settle down before proceeding to the next test.

EXPERIMENTAL RESULTS

Preliminary

In work of this nature, numerical results must be accepted with reserve. Gauge calibration for dry air is initially accurate to within about 5% of the reference gauge, and the check calibration after each test rarely differed significantly from the original one (see Appendix). We do not yet know the calibration for any pumping fluid vapour, (though we suspect the gauges are several times more sensitive in a vapour atmosphere than in air*.) Nor do we know whether

* For a discussion of this subject see 'Vacuum Technique' by S. Dushman, p. 349 et seq.

the gauge calibrations for the various vapours encountered differ significantly from each other, nor the effect of the presence of the gauge on the actual pressure.

Dry air calibration is used throughout, and where the atmosphere is largely vapour, results may be regarded as approximately comparative, the true pressures, being considerably lower than those recorded.

In an equipment so frequently dismantled and reassembled it was difficult to ensure leak tightness to the exacting standard desired. It is possible that slight air leakage sometimes contributed to the residual pressure after the vapour had been trapped out. The X-ray effect may also have made a significant contribution at very low pressures (see Appendix).

Conclusions can be drawn with more confidence from the consistent pattern of the results. The tests have continued for over 18 months and a great mass of observations has been recorded ; the same broad pattern of behaviour has been observed in each of about 20 runs and it is on this that the most interesting part of our conclusions is based.

(1) Pump-Down Curves

The curves in Fig. 4 all refer to the initial pump-down with fresh charges of fluid in the pumps. The ionization gauges were switched on about half an hour after the pump heaters, and readings were recorded from the time when the gauges had settled down.

The curves cover the first 30 hours of pump-down for the various fluids in the three pumps. No attempt has been made to correct to a standard cooling water temperature, but the approximate temperatures are indicated for each fluid. Two main conclusions can be drawn :

Firstly, each family of three curves for a particular fluid displays very clearly the marked difference made by different self-purifying arrangements. Pump C always ' conditions ' more rapidly and to a lower pressure than Pump B, which is in turn much superior to Pump A.

Secondly, though there is considerable variation in the ' conditioning rates ' of the different fluids, there is no general tendency in the cases of pumps B and C for the prepared pumping fluids taken as a group to ' condition ' more rapidly than the plasticisers similarly taken, or to produce lower ultimate pressures. In the case of Pump A, however, the purifying action is not so effective and the prepared fluids as a group do, on the whole, give better results than the plasticisers.

(2) Temperature—Pressure Curves

For this test the constant head apparatus and electric heater were connected to supply warm water at a number of different temperatures to each pump and header in turn. Each temperature was held steady until the ionization gauge reading had stabilized. The thermometer on top of the header was used to check whether header temperature differed significantly from water temperature, and when slight differences occurred the lower temperature was taken. Plots of Logarithm of indicated pressure against reciprocal of header temperature (°C Abs.) were obtained for each pump (Fig. 5).

The total vapour pressure in the header (assumed to be clean) should be the sum of the partial vapour pressures of the pumping fluid constituents, its impurities and its vapour decomposition products, the contribution of each depending on its relative abundance in the header*. Since backstreaming is continuous and the header is always the coldest part of the system, this total vapour pressure should be the *equivalent saturation vapour pressure* of the mixture at each header temperature.

There is also, of course, a permanent gas pressure in the header, which is not likely to be affected much by moderate changes in temperature.

The lower ends of the curves in Fig. 5 provide a means for comparing the ultimates obtainable with the three pumps and the various fluids, though in some cases there was a gradual improvement in ultimate throughout the rest of the test run.

The three curves obtained for any particular fluid in the three pumps show clearly the different abilities

* Hickman states that the contribution of the lighter constituents is unexpectedly large at low pressures[4].

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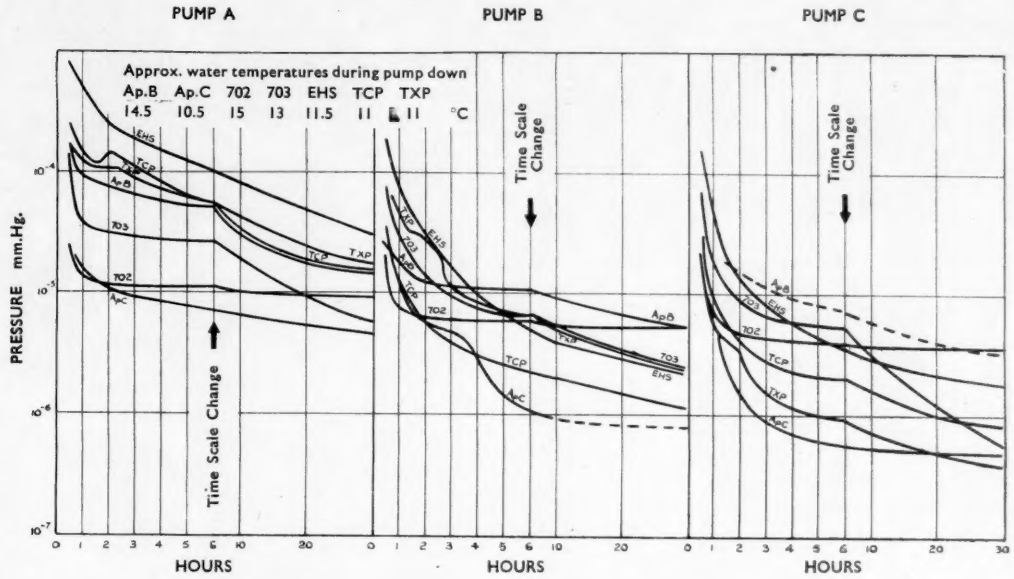
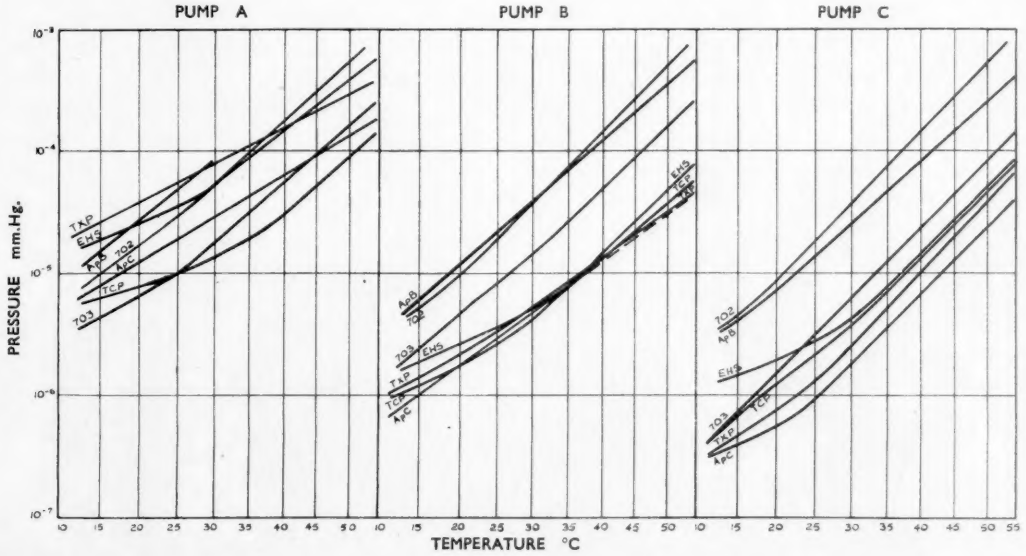


Fig. 4.—Pump-down curves for various fluids in pumps A, B and C. NOTE : During the run with Apiezon B pump C was switched off for over an hour to correct a fault. This has been allowed for in plotting the curve. When pump B was charged with Apiezon C some pressure fluctuation occurred in the dotted region but this ceased after about 35 hours pumping.

Fig. 5.—Temperature-pressure curves for various fluids in pumps A, B and C (the dashed line is extrapolated).



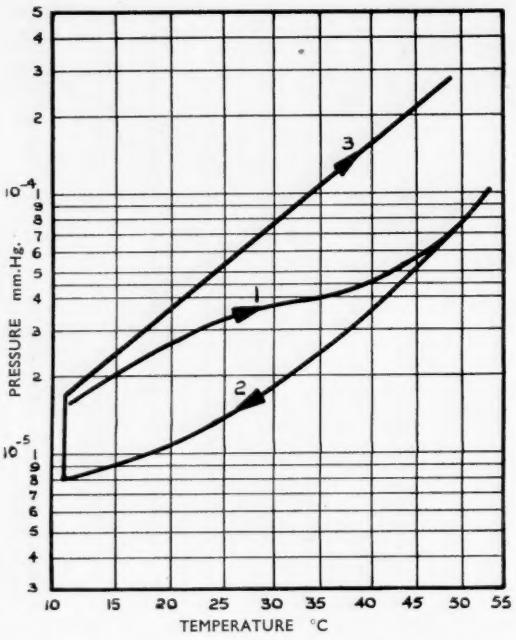


Fig. 6.—Temperature-pressure curves for Tri-Xylenyl Phosphate under varying conditions.

of the pumps to purify their fluid charges. The curve for Pump A always lies above that for Pump B, which is in turn above that for Pump C ; though in one or two cases the differences are not very great.

The plasticisers as a group are not inferior to the prepared pumping fluids when used in Pumps B and C, but when they are used in PumpA the disadvantages of initial impurity, etc., are not completely overcome and there is a tendency for group inferiority, considering of course the lower ends of the curves.

Since the vapour in the headers is saturated the curves, as plotted, should be approximate straight lines provided two further conditions are fulfilled :

(1) The partial pressure of vapour in the chamber is large compared with the partial pressure of permanent gas.

(2) The composition of the vapour does not change and the vapour remains saturated.

The permanent gas pressure is probably largely exerted by gaseous decomposition products and by air from small leaks. If at the lower temperatures the gauge deflection due to vapour becomes comparable with that due to gas, then the lower end of the pressure-temperature curve will bend towards the horizontal. This tendency is exhibited by several of the curves.

Curvature or distortion of the curves may also be due to changing vapour composition as temperature is raised. This sort of distortion certainly occurred when the curve was taken for Tri-Xylenyl Phosphate in Pump A. The effect is illustrated in Fig. 6.

Line 1 in Fig. 6 was taken as the water temperature was raised in steps ; the line seemed to shift to the right during the test, so that pressures were much lower than was expected at the high temperature end. Line 2 was obtained by dropping the temperature in steps after taking Line 1 ; it has a normal slope and so lies well below Line 1 at the low temperature end. After a period of waiting with normal cooling flow and temperature, the pressure rose to its initial value. The side tube cooling turn was then fed with a separate water supply which remained at mains temperature, and Line 3 was taken ; this is reasonably straight and contains points taken with rising and falling temperature.

Water flow is considerably reduced during these tests, and, since heat is supplied to the water by the pump, the side tube cooling turn runs unusually warm. As a test proceeds and the water supply is itself warmed the side tube temperature increases further. It is apparent that the pumping fluid was being progressively purified during the taking of Line 1 and remained in the improved condition during the taking of Line 2, only returning to normal after full normal cooling had been restored. During the taking of Line 3, the efficiency of the side stage as a purifier was kept constant, so the fluid composition did not change.

This effect appeared to be considerable only in the case of Tri-Xylenyl Phosphate, so the other curves presented for Pump A were all obtained with a single water supply.

(3) Freezing Out the Vapour

For this test the cooling flow to each pump was arranged to by-pass the header, and each header was

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strongly cooled by heaping crushed solid carbon-dioxide on the top and in the trough (see Fig. 3). Thermometers in the top and side pockets registered about —30° to —50°C.

The drop in ionization gauge indication after applying the coolant is assumed to represent the vapour component of the pressure existing in the water cooled header. The residual pressure recorded after cooling is likely to be largely a gas pressure supplied from fluid decomposition and from small leaks, etc., possibly combined with a spurious pressure reading due to the 'X-ray effect' (see Appendix). There may still, however, be some vapour contribution for vapour may beam up from the baffle in the pump mouth (which is not strongly cooled) and affect the gauge before being condensed.

The freezing-out was done partly to make sure that air leakage was not too seriously affecting our results, but other inferences can be drawn from the pressures obtained (see Table 1).

In the cases where the pressure obtained after cooling is small compared with the pressure before cooling, the temperature-pressure curves discussed in the last section are likely to be relatively straight. This is illustrated by the cases of Silicone 703 in Pump C and Silicone 702 in Pumps B and C.

In the cases where the pressures obtained after cooling are more comparable with those obtained before, the temperature-pressure curves are likely to have some curvature. This is illustrated by the cases of Silicone 703 in Pump A and by all the Di-Ethyl Hexyl Sebacate curves. These curves can be considerably straightened out by subtracting the 'permanent gas' pressures obtained after cooling from the ordinates at all temperatures. The curves then more truly represent the variations of vapour pressure with temperature in the headers. The fact that over-compensation never occurs when this subtraction is made suggests that there is no considerable vapour component in the residual pressure after freezing-out.

A very important feature of the results in Table 1 is the regularity with which the residual pressure above Pump C is lower than that above Pump B which is, in turn, lower than that above Pump A. The differences are considerable and the effect is too consistent to be due to the accident of leakage.

It is apparent from results to be discussed later that the higher residual gas pressures recorded for Pump A are largely supplied from thermal decomposition of the fluids. Gases cannot accumulate in a boiler as can liquids. The actual rate of gas evolution must be greater in Pump A than in the other pumps. It seems likely that a large part of the gas comes from the breakdown of intermediate decomposition products and that the more rapid removal of these products from Pumps B and C has an important influence on their rate of gas evolution.

Slight leakage and spurious effects may have contributed to increase some of the pressures recorded, so too much significance should not be accorded to precise numerical values. However, the general purport of the results is obvious and very important. Pump A is not inferior to many oil pumps in current use. Such a pump, even using a highly purified fluid and a refrigerated trap, may be incapable of producing a pressure as low as 10^{-6} mm. Hg. It is apparent why some workers whose experience of oil pumps may be limited to types comparable with Pump A, still prefer to use mercury pumps where the very lowest gas pressures are desired. Mercury cannot decompose.

Table 1 **Pressure Changes when Headers are Strongly Cooled (mm. Hg)**

FLUID	PUMP A	PUMP B	PUMP C
Silicone 702	$2.3 \times 10^{-5} \rightarrow 3.2 \times 10^{-6}$	$7.5 \times 10^{-6} \rightarrow 7.2 \times 10^{-7}$	$4.2 \times 10^{-6} \rightarrow 2.25 \times 10^{-7}$
Silicone 703	$4.2 \times 10^{-6} \rightarrow 1.57 \times 10^{-6}$	$2.5 \times 10^{-6} \rightarrow 9.8 \times 10^{-7}$	$5.2 \times 10^{-7} \rightarrow 5.0 \times 10^{-8}$
Apiezon C	$5.2 \times 10^{-6} \rightarrow 1.4 \times 10^{-6}$	$1.35 \times 10^{-6} \rightarrow 4.5 \times 10^{-7}$	$3.2 \times 10^{-7} \rightarrow 1.3 \times 10^{-7}$
Tri-Cresyl Phosphate	$7.4 \times 10^{-6} \rightarrow 5.3 \times 10^{-6}$	$8.25 \times 10^{-7} \rightarrow 1.65 \times 10^{-7}$	$7.8 \times 10^{-7} \rightarrow 8.8 \times 10^{-8}$
Tri-Xylenyl Phosphate	$1.65 \times 10^{-5} \rightarrow 3.75 \times 10^{-6}$	$1.4 \times 10^{-6} \rightarrow 2.5 \times 10^{-7}$	$3.15 \times 10^{-7} \rightarrow 6.5 \times 10^{-8}$
Di-Ethyl Hexyl Sebacate	$1.2 \times 10^{-5} \rightarrow 8.8 \times 10^{-6}$	$2.0 \times 10^{-6} \rightarrow 1.35 \times 10^{-6}$	$1.0 \times 10^{-6} \rightarrow 6.0 \times 10^{-7}$

One further result of the test was to establish that any X-ray effect correction must be less than 5×10^{-8} mm. Hg, the lowest pressure recorded.

(4) The Conversion of Pump A to Resemble Pump B

Pump A differs from Pump B in having an extra cooling turn round the side tube and in having a greater heater input.

In this test the extra cooling turn on Pump A was by-passed and allowed to drain dry, and gauge readings were recorded against time until they had become substantially steady after the change. The heater input of Pump A was next reduced to match that of Pump B so that the two pumps were identical.

side jet. They are presented for certain fluids in Table II.

By-passing the cooling turn always produced an immediate improvement in fine pressure which continued to fall steadily for several hours after the change to a final value between about a half and about a thirtieth of the original. Reducing the heater input usually produced a further, though less marked, improvement in fine pressure.

A return first to normal heater input and later to normal cooling flow produced successive deteriorations in fine pressures to a final value of the same order as the original one, though always somewhat lower. Not all the improvement was lost.

It is apparent that the drop in fine pressure on by-

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Table II **Results of Modifying the Cooling and the Heater Input of Pump A so that it Resembles Pump B**
(Pressures in mm. Hg)

FLUID	INITIAL PRESSURE	SIDE STAGE UNCOOLED	INPUT* REDUCED	FULL INPUT RESTORED	SIDE STAGE RE-COOLED	CORRESPONDING PRESSURE FOR PUMP B
Silicone 702	10^{-5}	6×10^{-6}	5.2×10^{-6}	6×10^{-6}	8×10^{-6}	5.8×10^{-6}
Silicone 703	3.05×10^{-6}	1.8×10^{-6}	1.8×10^{-6}	1.9×10^{-6}	2.6×10^{-6}	1.8×10^{-6}
Apiezon C	3.8×10^{-6}	1.05×10^{-6}	8×10^{-7}	1.1×10^{-6}	2.7×10^{-6}	1.0×10^{-6}
Tri-Cresyl Phosphate	1.3×10^{-5}	8.1×10^{-7}	6.5×10^{-7}	8×10^{-7}	1.05×10^{-5}	6×10^{-7}
Di-Ethyl Hexyl Sebacate	1.2×10^{-5}	2.3×10^{-6}	1.85×10^{-6}	2.2×10^{-6}	1.2×10^{-5}	2.05×10^{-6}

*Giving full correspondence with Pump B c.f. last column.

Gauge readings were again recorded against time.

The normal heater input was next restored to Pump A, and gauge readings were noted, and finally the extra cooling turn was restored and gauge readings were noted until they had again become steady.

It has been explained how the presence of the extra cooling turn round the side tube may reduce the ability of Pump A to purify its working fluid. The extra heater input to this pump may also affect its performance by increasing the decomposition rate in the boiler.

The change in heater input was made at this stage to complete the correspondence of Pump A to Pump B. More complete observations of the effects of heater input variation were made in a separate test.

The results of the test are striking and provide some of the best evidence about the purifying action of the

passing the cooling turn is due to the better elimination of impurities and decomposition products from the pump and that the further improvement on reducing the heater input is due to a reduced decomposition rate in the boiler.

The deterioration of the fine pressure to nearly its initial value, on restoring the original conditions, must then be due to the concentration of decomposition products in the pumping fluid increasing until they can be thrown out by the cooled side stage as rapidly as they are produced.

The pressures did not return completely to the initial values. It would seem that some impurities were initially present which were not the result of fluid decomposition. If a proportion of these was thrown out by the hot side stage, their concentration in the pump would be permanently reduced.

The magnitude of the effects described would be expected to vary with the chemical stability of the fluid. The pressure changes were in fact least marked with the supremely stable silicone fluids and most marked with the plasticisers.

The ultimates obtained by the modified Pump A agreed satisfactorily with those obtained by Pump B.

(5) Variation of Heater Input

In this test, the supply voltage for the three pumps (normally 230 volts) was set at each of a number of different values in turn, and at each setting the pressures were recorded.

Though a change in heater input to a pump produces only a small change in boiler temperature (see Fig. 2), this may cause a comparatively large change in the decomposition rate of the fluid, and it is likely that the concentration of decomposition products—and with it the ultimate pressure—will increase with rising boiler temperature and vice versa.

The fine pressure, then, should fall with heater input until the jets become insufficiently dense to prevent back diffusion, thereafter the fine pressure will rise with reducing input.

The curves in Fig. 7 display the general tendency described above, though the input has not always been reduced low enough to show the minimum for all three pumps.

The comparative flatness of the curves for Pump C is of interest. Some of the curves for this pump (*e.g.* those taken with Silicone 702 and Tri-Xylenyl Phosphate) are completely flat over a considerable range of inputs. A possible explanation is that, for some fluids, the level of decomposition products in this pump is kept so low that their contribution to the chamber pressure is small and so changes in their abundance do not much affect it.

To check this idea, some tests were made with Silicone 702 in the three pumps. Silicone 702 is probably the most stable diffusion pump fluid available (excluding Mercury). Its only rival is Silicone 703, but this fluid boils at a higher temperature in the boiler and is therefore likely to decompose faster (see Fig. 2). It should be easier to obtain low concentrations of decomposition products with Silicone 702 than with any other fluid.

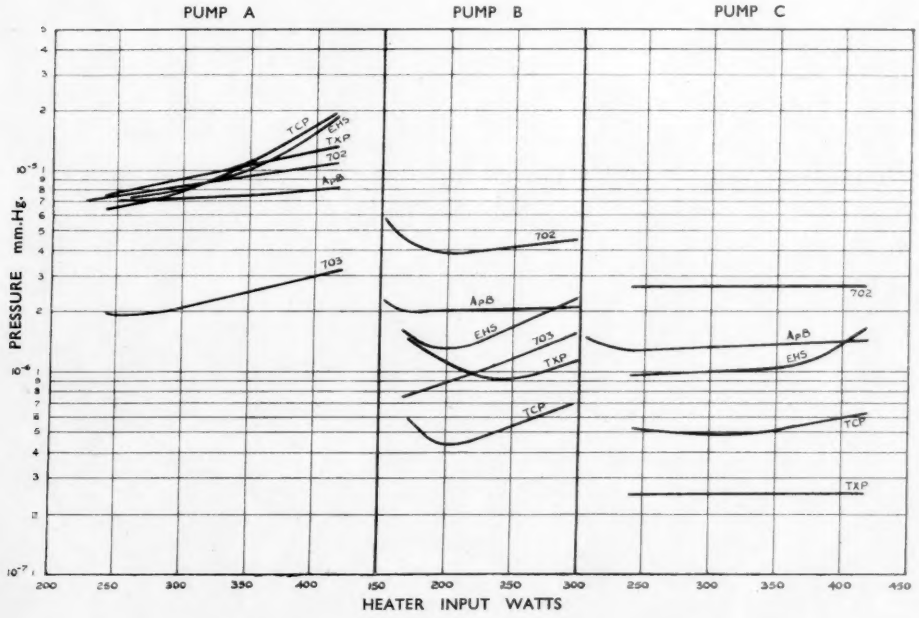

Fig. 7.—Fine pressure-heater input curves. NOTE: All pressures corrected to a nominal header temperature of 10°C.

The fine pressure-input curves with this fluid were taken over an extended range of inputs and at three backing pressures : 0.3, 0.02 and about 0.001 mm. Hg. They are presented in Fig. 8.

The three curves for Pump C are all flat and register approximately the same pressures until the inputs become so low that the pump breaks down. We suppose this to indicate that the concentration of decomposition products is small.

The three curves for Pump B are all comparatively flat at inputs below normal and above ' breakdown ', and the flat portions of the curves register approximately the same pressure as do the curves for Pump C ; there is some upward curvature at inputs above normal in the case of the curve at high backing pressure and the curve at low backing pressure is more truly flat than the other two and registers a somewhat lower pressure than do they. We suppose this to indicate that at normal input and below it the concentration of decomposition products is quite small, though a high backing pressure may hinder the sufficiently rapid removal of decomposition products at higher inputs, and a very low backing pressure may accelerate their removal.

The two curves for Pump A taken at 0.02 and 0.3 mm. Hg backing pressure slope strongly downward with reducing input till ' breakdown ' is reached. We conclude that the concentrations of decomposition products are considerable, but decrease with decomposition rate on reducing the input.

When the 0.001 mm. Hg backing pressure is applied to Pump A, the improved distillation of decomposition products into the backing line causes a considerable drop in fine pressure before input variation is begun. Moreover, at this low backing pressure the input, and with it the decomposition rate, can be reduced very substantially before breakdown and a flat curve can be obtained over a range of inputs. Over this range the curve registers approximately the same pressure as did the flat curves for the other pumps. Flatness has been achieved by better elimination of decomposition products and by reduction of decomposition rate, and we conclude that a low level of decomposition products is one not unlikely explanation for a flat fine pressure-input curve.

The approximate correspondence of fine pressures achieved by the three pumps over the flat portions

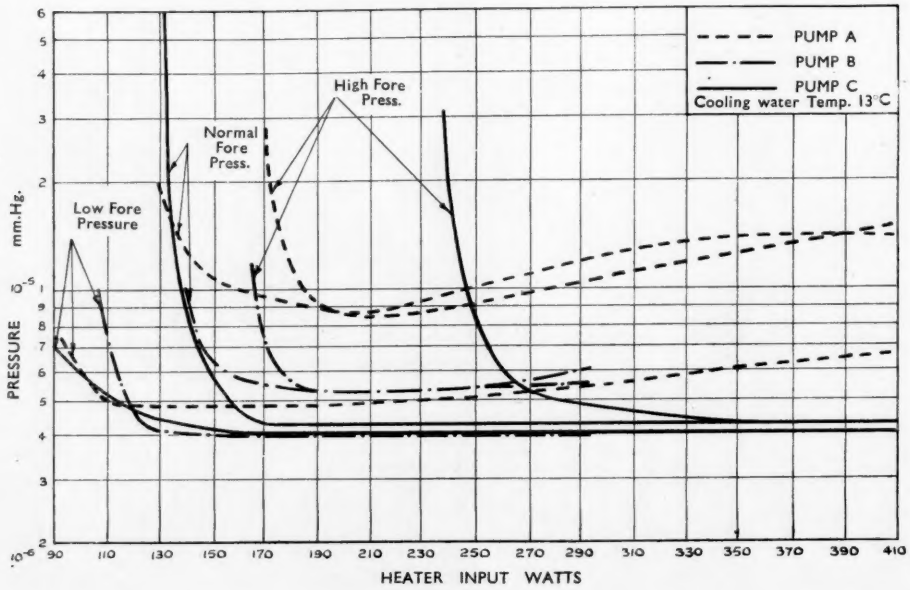

Fig. 8.—Fine-pressure heater input curves for Silicone 702 at various fore pressures.

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of the curves is further evidence that, under these conditions, an approximately pure fluid has been obtained in each pump so that each header contains substantially pure Silicone vapour. The pressures recorded are quite compatible with this view.

Decomposition contributes a gas, as well as a vapour component to the fine pressure. A reduced level of decomposition products should correspond to a reduced permanent gas pressure in the header. When a header is strongly cooled, the vapour should be substantially condensed and the residual pressure should be permanent gas.

When Silicone 702 vapour was condensed out of the header of Pump C (Table 1) the pressure dropped from 4.2×10^{-6} to 2.25×10^{-7} mm. Hg, roughly a 20 : 1 improvement.

(6) Variation of Backing Pressure

Backing pressures were first reduced to below one tenth of the normal by switching on the backing diffusion pump, and then increased to the order of ten times the normal by opening the needle valve. Variations in fine pressure were noted. The results are presented for some of the fluids listed in Table III. They can be summarized as follows :—

The fine pressure of Pump C is comparatively insensitive to backing pressure variation. With no fluid did the fine pressure change by as much as 50% over the full range of backing pressure variation. With some fluids there was no change at all.

Pump B is also insensitive to backing pressure, though there was recorded with the fluid Di-Ethyl Hexyl Sebacate a total fine pressure change of just over 100% over the full range of backing pressure variation.

Pump A is scarcely more sensitive than Pump B to backing pressure increase, but backing pressure reduction does often bring a considerable improvement in fine pressure. The improvement is gradual and is probably due to the easier distillation of impurities and decomposition products into the backing line.

In general, though the highest backing pressure used was 300 times the lowest, fine pressure changes were comparatively small, especially for Pumps B and C. We conclude that, with the three pumps tested, back diffusion through the pump and the solution of air in the condensed working fluid make no considerable contribution to the fine pressure.

The fine pressure changes which do occur are chiefly associated with Pump A, and seem likely to be

Table III **Fine Pressure Changes when Backing Pressure is Varied (mm. Hg)**

	SILICONE 702			APIEZON C			DI-ETHYL HEXYL SEBACATE		
Backing Pressure	A	B	C	A	B	C	A	B	C
Normal (~0.02)	1.25×10^{-5}	5.4×10^{-6}	4.0×10^{-6}	2.7×10^{-6}	7.2×10^{-7}	3.2×10^{-7}	1.2×10^{-5}	1.2×10^{-6}	1.1×10^{-6}
High (~0.3)	1.4×10^{-5}	5.4×10^{-6}	4.2×10^{-6}	2.6×10^{-6}	7.2×10^{-7}	3.2×10^{-7} *	1.85×10^{-5}	2.4×10^{-6}	1.4×10^{-6}
Low (~0.001)	5.5×10^{-6}	4.4×10^{-6}	4.0×10^{-6}	2.7×10^{-6}	7.2×10^{-7}	3.2×10^{-7}	4.4×10^{-6}	1.0×10^{-6}	1.0×10^{-6}

*Taken at 0.1 mm. Hg. Pump C with Apiezon C broke down at about 0.3 mm. Hg.

Pump A was now set at its normal input and backing pressure (0.02 mm. Hg) and the freezing-out test performed on it. The pressure fell from 7.8×10^{-6} to 9.2×10^{-7} mm. Hg, an improvement of 8.5 : 1. The coolant was removed and the input set to 150 watts and the backing pressure to 0.0018 mm. Hg—conditions corresponding to the flat part of the fine pressure-input curve. When conditions had become steady the freezing out test was repeated and this time the pressure fell from 5×10^{-6} to 2.4×10^{-7} mm. Hg, an improvement of more than 20 : 1 and in very close agreement with the results for Pump C.

Here is clear evidence of the contribution which decomposition may make to the gas as well as to the vapour component of the fine pressure, even with a very stable fluid, and further proof that Pump A is comparatively free of decomposition products when working on the flat part of the curve.

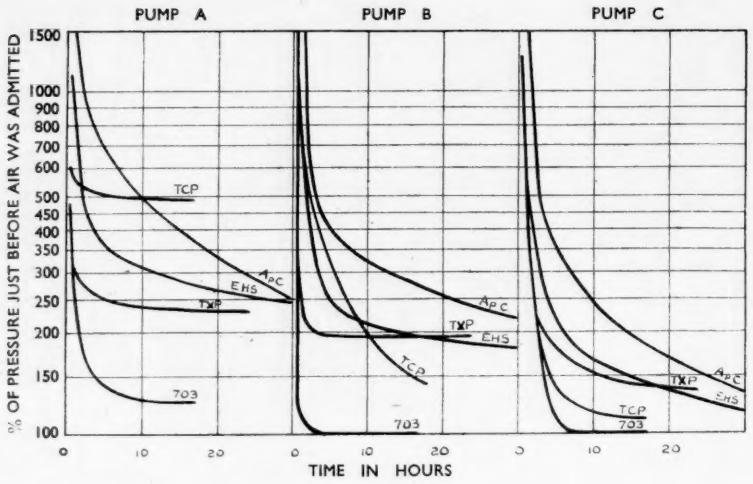

Fig. 9.—Pump-down curves after exposure to atmosphere of hot fluids. NOTE : In the case of Silicone 703 in pump C the gauge was switched on 30 minutes later.

due to the influence of backing pressure on the distillation of impurities from the side stages into the backing line.

(7) The Exposure of Hot Pumping Fluid to Air at Atmospheric Pressure

The pump heaters, the gauges and the rotary backing pump were switched off, and the air admittance valve in the backing line was immediately opened. When the hot pump fluid had been exposed to atmosphere for ten minutes, the air admittance valve was closed and all the pumps were switched on. The gauges were switched on a few minutes later, and pump-down curves were taken as in the first test described (Fig. 9).

To enable Fig. 9 to illustrate comparative recovery times, irrespective of the other qualities of pumps and fluids, pressures for each combination of pump and fluid are plotted as percentages of the pressure that combination was producing just before air was admitted. To reach the 100% line means full recovery —whatever the initial ultimate of the particular combination.

The results were much as expected. Silicone 703 recovered rapidly to give the original order of vacuum in all three pumps a few hours after exposure.

Apiezon C took about 20 times as long as the Silicone completely to recover in Pump C and even then recovery was not complete in Pumps A and B, but was probably continuing.

The plasticisers behaved much as Apiezon C in Pumps B and C, though they were perhaps a little faster in recovery. These fluids in Pump A were generally worse than the Apiezon fluid, Tri-Cresyl Phosphate being particularly bad.

The tests again illustrate the exceptional stability of the Silicone fluids and the merits of Pumps B and C. All the fluids, given time, would recover completely from accidental exposure in Pump C.

(8) Ultimate Pressures

In some cases the ultimates continued to improve after the temperature-pressure curves were taken. Table IV presents the lowest pressures recorded with

Table IV

Ultimate Pressures (mm. Hg.)

FLUID	PUMP A	PUMP B	PUMP C	APPROX. HEADER TEMP °C.
702	9.8×10^{-6}	5.4×10^{-6}	4.0×10^{-6}	13
703	3.5×10^{-6}	2.0×10^{-6}	3.6×10^{-7}	13
Ap. B	1.4×10^{-5}	5.3×10^{-6}	2.5×10^{-6}	15
Ap. C	3.4×10^{-6}	$\sim 6 \times 10^{-7}$	3.2×10^{-7}	11
TCP	$\sim 1 \times 10^{-5}$	6.1×10^{-7}	3.2×10^{-7}	11
TXP	1.2×10^{-5}	9.7×10^{-7}	2.7×10^{-7}	11
EHS	1.2×10^{-5}	1.7×10^{-6}	9.2×10^{-7}	11

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the pumps working normally. The pressures for Pumps B and C were taken near the ends of the runs, but before Test 7. Those for Pump A were taken before Test 4, for this test usually produced a permanent improvement in the condition of the fluid.

CONCLUSIONS

(1) The Pumps

The three pumps appear to work substantially in the manner described in the first sections of the paper.

Comparing the rates of pump-down and the ultimate fine pressures reached, Pumps B and C are much superior to Pump A. Pump C always pumps down faster with a new fluid filling and to a lower ultimate pressure than does Pump B. The degree of difference varies with the fluid used. Pump C recovers fastest from exposure to air when hot.

Pumps B and C are capable of producing useful fine pressures with all the plasticisers tested, and purify them so effectively that in two cases these fine pressures are better than those obtained with some of the prepared fluids.

These results are obtained with a relatively simple metal construction and without prejudice to speed and backing pressure performance. The speeds of all these pumps (all of $2\frac{1}{4}''$ bore) are of the order of 70 litres/sec. and the limiting backing pressures are of the order of 0.5 mm. Hg, though these figures vary somewhat with the fluids used.

Self-purification is an effective alternative to fractionation in suitable pumps and is probably more effective than fractionation alone in small pumps where considerable fluid decomposition is encountered.

(2) The Fluids

Comparing the ultimate pressures reached with the various fluids, Apiezon C, Silicone 703 and Tri-Cresyl and Tri-Xylenyl Phosphate all gave pressures of the order of 5×10^{-7} mm. Hg in Pump C and there is little to choose between these four fluids in this respect when they are used in Pumps B and C.

With Pump A, the purity and chemical stability of the fluid become more important, and Tri-Xylenyl Phosphate gave a pressure about ten times that given by Silicone 703 when the fluids were compared in this pump.

Di-Ethyl Hexyl Sebacate was a little disappointing, though quite useful ultimates were obtained with it in Pumps B and C. It is possible that some of the construction materials severely catalysed its decomposition.

We have little experience in the use of the plasticisers for routine pumping and we would hesitate at present to recommend their adoption. They can obviously only be used in suitably designed pumps and they are obviously much less well able than the Silicones to stand up to rough conditions and accidental exposure. We have used Tri-Cresyl Phosphate for routine vacuum coating work in this laboratory in three 9″ bore pumps of a design similar to type B. One pump was in regular use for about six months. No unusual difficulties were experienced.

(3) The Ultimate Fine Pressure and Fluid Decomposition

Perhaps the most difficult problem which the pump designer has to face in building a commercial pump to give a very low ultimate pressure is the thermal decomposition of the pumping fluid. The other factors mentioned in the introduction to this paper do not seem to be very troublesome if the design is competent.

We suspect that both the vapour and gas ultimates of many pumps in industrial use are affected by fluid decomposition to a far greater extent than is realized ; so that the vapour pressure in the header corresponds to that of a much more volatile mixture than the pure pump fluid would provide, and the total pressure cannot be reduced as much as might be expected by refrigeration.

Ideal conditions and arrangements to reduce the concentration of decomposition products in the header have been discussed by others, notably by Hickman, but they are difficult to provide in a simple rugged construction.

The authors wish to thank Mr. Barrett, the Technical Director of W. Edwards & Co. (London) Ltd., for permission and encouragement to make these investigations and to arrange for their publication.

Appendix

Gauges, Control Units and Gauge Calibration

Because it is so difficult to compare the results of different investigators unless gauging methods are similar, we add a short description of our gauges and calibration technique.

The electrode assemblies consisted of a renewable tungsten filament, a molybdenum wire grid and a pair of nickel plates, all carried on nickel supports set in a glass pinch. The dimensions were similar to those of the gauge described by E. K. Jaycox and H. W. Weinhart[6]. This assembly was freely supported in a test header as already described.

The control unit provided stabilized potentials of +100V for the grid and —25V for the plates with respect to the filament. It supplied a current to the filament such that the emission was kept constant at 2 or 5 mA. The plate current was measured by an electronic microammeter.

When zeroing the microammeter, the filament was switched off and a resistance was connected externally to carry from the filament to the grid a current equal to the normal emission. Under these conditions the control unit maintained the normal potentials on all the electrodes and any leakage current to the plate was balanced out in the zeroing.

The gauges were calibrated before and after use against a high compression McLeod gauge (compression ratio ~2400 : 1 per cm.). Cold traps were used in the McLeod line and above the pump and dry air was admitted below the latter cold trap during calibration. Readings were taken over the pressure range 10^{-4} to 5×10^{-3} mm. Hg. and a calibration factor was obtained from the resulting straight line.

No special outgassing methods were used on the gauges during calibration or during use. Pumping was merely continued until the readings were steady.

Dayton[7] has stated that the sensitivity of ionization gauges used with Silicones may decrease many times during use, due to the formation of an insulating film on the ion collectors, so that pressures are recorded many times lower than are actually obtainable with these fluids. We therefore present a table of the calibration factors obtained for the gauges, before and after use with some of the fluids (a test run usually lasted over 350 hours, during over 100 hours of this time the gauges were switched on). We find no evidence for Dayton's view and we can only suggest that gauges with platinum plates such as he used may catalyse fluid decomposition to a much greater extent than do gauges of the type we used.

Table V

Calibration Constant C for Ionization Gauges before and after Use

Pressure mm. Hg = C × 10^{-5} × (ion current μA) when electron current is 2 mA.

FLUID	PUMP A		PUMP B		PUMP C	
	Before	After	Before	After	Before	After
702	9.0	9.0	8.0	8.0	9.0	—
703	7.0	8.0	8.2	8.2	8.2	8.2
Ap. B	7.6	8.0	8.2	8.2	8.2	—
Ap. C	6.75*	8.0	9.0	9.0	8.6	8.6
TCP	7.4	9.6	7.8	8.4	7.8	7.8
TXP	9.2	9.2	9.0	9.0	9.0	9.0
EHS	8.9	8.9	8.6	8.6	8.0	8.4

*This first calibration was considered inaccurate since the points were very divergent.

Metson[8], among others, has found that a spurious plate current may be recorded with an ionization gauge because electrons are emitted from the plate due to bombardment by soft X-rays from the grid. This current would set a lower limit to the pressure it is possible to record, even if absolute zero pressure was actually obtained. With the electrode assemblies and potentials we used we believe this lower limit is roughly of the order of 10^{-8} mm. Hg.

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2 JAECKEL, R., *Z. Tech. Phys.*, (1942), 177-186.
3 HICKMAN, K. C. D., *J. Appl. Phys.*, **11**, (May, 1940), 302.
4 HICKMAN, K. C. D., *Rev. Sci. Instrum.*, **22**, (March, 1951), 141.
5 BLEARS, J., *Proc. Roy. Soc.* **188A.**, (Dec. 31, 1946), 62.
6 JAYCOX, E. K., & WEINHART, H. W., *Rev. Sci. Instrum.*, **2**, (1931), 401.
7 DAYTON, B. B., *Le Vide.*, **2**, (Nov., 1947), 349.
8 METSON, G. H., *Brit. J. Appl. Phys.*, **1**, (March, 1950), 73 and **2**, (Feb., 1951), 46.

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LETTERS TO THE EDITOR

Notes on the Coating of Plastic Foils

Sommaire

L'AUTEUR COMMENTE l'article de Mr. Holland et la lettre du Dr. Zehden, tous deux publiés dans le numéro de janvier. Il ajoute l'observation que des films plastiques ont souvent tendance à s'étirer et à se déchirer par suite de la chaleur radiante de la source d'évaporation et donne connaissance des mesures à prendre pour réduire sensiblement cet inconvénient.

I HAVE READ with great interest the article by Mr. L. Holland entitled "The Production, Properties and Uses of Thin Films Condensed in Vacuo".

Depositions by thermal evaporation or positive ion bombardment in a vacuum chamber are an essential and much used service of the Electronics Division at this Establishment. We have experienced very similar results to those reported by Mr. W. Zehden in your correspondence columns, especially when using masks in order to coat selected portions of targets. We find it essential to use very thin stencils in intimate surface contact with the specimen, which latter must either be really cold or outgassed before deposition begins. Boundary or edge-effects have been particularly noticeable with coatings of bismuth on aluminium discs, and with aluminium on paper or 'plastic' sheets.

Thin 'plastic' films when coated by evaporation methods very often stretch and tear due to radiant heat from the source. This hazard can be substantially reduced by the following processes :—

(a) mounting the target on a relatively large block of copper to conduct heat away.
(b) by precoating with a semi-reflecting coat of a metal with a low vaporisation temperature.
(c) the use of plaited or twisted wires in the preparation of the conventional flat "sinusoidal" heater coil, or if a single wire is essential the bell-jar geometry must be arranged so that the coil is mounted in a horizontal plane.

The reason for the last process has been demonstrated by Strong in 'Procedures for Experimental Physics' on page 174. The use of plaited wire will usually allow complete surface 'wetting' of the source especially when evaporating light metals. If the wire is solid and mounted vertically, 'pinhole self-photographs' will show that the top loops of the coil are not wetted and only contribute harmful radiant heat to the fragile target.

In the horizontal plane there appears to be no disadvantage in using a single wire coil.

The above remarks, of course, do not apply when using rhodium, which is conventionally deposited by sublimation from an electroplated source.

Acknowledgement is made to the Director of the Atomic Energy Research Establishment for permission to publish these notes.

T. A. J. Jaques.
Electronics Division.
Ministry of Supply,
Atomic Energy Research Establishment,
Harwell,
Didcot,
BERKS.

4th April, 1951.

Production of Small Apertures by an Evaporation Technique

Sommaire

DEUX MÉTHODES pour percer des petits trous (diamètre approximatif 3 microns) dans du feuillard de quelques milliers d' Å d'épaisseur, sont décrites. La première produit un dépôt ombré autour d'une goutte d'huile, mais est difficile, particulièrement en ce qui concerne le montage de tels films. La seconde méthode a donc été développé. La plaque de verre est recouverte d'une couche détachable, sur laquelle repose une aiguille polie. L'évaporation se fait d'une source entourant l'aiguille, donnant un film pratiquement uniforme autour de la pointe. L'auteur donne des détails de la préparation de l'aiguille et de montage du film. Cette méthode n'est pas limitée à la production de trous isolés, mais permet la production en masse.

IN CONNECTION with experiments on Gabor Diffraction Microscopy [1,2] a demand arose for a number of small holes of approximately 3 microns in diameter in metal foil as thin as possible whilst still remaining opaque to light, *i.e.*, of the order of a few thousand angstroms. Such a hole possesses the characteristics of a true aperture in that the diameter of the hole is an order of magnitude greater than the thickness of the film

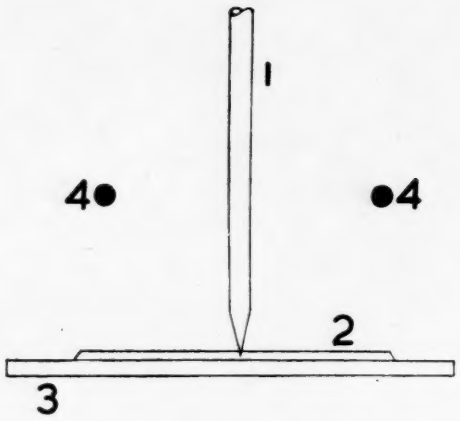

Fig 1

in which it is formed. Two methods have been used to produce such apertures.

(1) Metal is evaporated from a point source to the required average thickness about an oil drop sprayed on to a glass surface previously coated with a stripping layer for the purpose of removing the final film. This method is essentially a shadowing technique similar to that used in electron microscopy. Unless extreme precautions are taken it is difficult to avoid a penumbra effect in such work, with the result that the apertures, although frequently circular in form, normally possess a semi-transparent edge. Rapid formation of the film is required in order to avoid raising the vapour pressure of the oil by radiant heat from the evaporating source. The penumbra is enhanced, and the aperture formed normally somewhat larger than the drop, if heating is allowed to occur. The success of the method depends, to a large extent, on factors which are difficult to control and the rate of production of satisfactory apertures is low.

A further difficulty arises in the mounting of such films. In the thicknesses normally produced (approximately 2000 Å) it is difficult to mount successfully across an aperture exceeding a few millimeters in diameter. Thus it is necessary to achieve a fairly precise location of the hole before attempting the mounting procedure.

(2) These difficulties have been overcome by the alternative method indicated schematically in Fig. 1. Here an electrolytically pointed and polished needle (1) rests gently on the stripping layer (2), coated on the clean glass surface (3). Evaporation from the ring source (4) indicated about the needle, deposits a substantially uniform film around the point. The needle assembly is removed, and replaced by a rod provided with a cutting edge. The precise location of this method enables the tool to scrape a broad circular track through the film about the hole as centre. Excess film is removed with a razor blade and the desired portion raised from the glass surface with a few drops of water. A mounting ring in the form of a small polished washer, of external diameter equal to that of the film, is then inserted into the drop. Holes of this magnitude cannot be seen when on the water surface but the edge of the circular film allows the position to be determined with sufficient accuracy without direct reference thereto. The surface tension of the drop helps to maintain symmetry and, on evaporation of the water, the required hole is readily located in the aperture of the mounting ring.

During the evaporation of the water, the surface tension also causes a slight depression of the film in the ring aperture so that, when dry, a slight concavity exists. This has been found to be desirable in that cracks which frequently start from the hole in tightly stretched films are thereby avoided. For cases where this concavity is objectionable, a two-stage procedure is adopted. The film is first mounted as above on a ring somewhat larger than that finally desired, and is then transferred to the latter after drying.

The needles were prepared in the following manner from available 2 mm. diameter stainless steel rods. For a distance of roughly 1 cm. from one end, the diameter was reduced to approximately 0.25 mm. and this ground portion then further reduced and polished by a pointing method similar to that described by Grundfest et al[3]. Needles thus prepared may possess diameters of a few microns and be essentially parallel for one or two millimetres from the end (for films of the thickness considered here this is not, of course, a necessary requirement). The thick portion of the needle facilitates ease of handling while the short length of the required section still enables it to be mounted as shown in Fig. 1. By evaporating at a low angle to the stripping surface, shadow effects are avoided. The microscopically sharp end of the thin portion penetrates the soft stripping layer and is somewhat blunted by the pressure against the glass surface. No difficulty has arisen from this cause however. Indeed the effect appears to serve a very useful purpose in preventing difficulties caused by vibration.

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Developments in electron microscopy have led to the introduction of a variety of methods for producing hydrophilic stripping layers of the type required here[4]. Although not always necessary, a stripping layer is essential when films of the order of 1000 Å thick are to be removed from the glass surface. Clearly it is also necessary to pay careful attention to the preparation of this layer, for only when this is done will the final film be free of fortuitous pin holes—an obvious requirement. For this work a fairly thick layer of soap solution, prepared and applied in a dust-free atmosphere, has been found satisfactory.

The method is not limited to the production of one aperture per evaporation; indeed mass methods may be used throughout. Many needles may be shaped and polished in a single operation. By mounting these as a linear array, and evaporating simultaneously from both sides, many apertures may be produced economically.

The choice of metals is naturally wide. Silver, gold, aluminium, nickel, etc. are all possible. It should be noted that when aluminium is used it is desirable to make the initial film somewhat more than opaque so that this property is retained on subsequent oxidation.

The smallest aperture made to date was approximately 1 micron in diameter. Experience has shown that extension of the method to still smaller sizes is limited only by the method of mounting the needle. With the present method, the limit is reached when the flattening of the point exceeds the required hole diameter. Nor is the method limited to such films as described above. Films of up to 0.001 inch thick with 3 micron diameter holes have been made in aluminium, but the process is wasteful in material unless many are made simultaneously.

It is of interest to note that there is no difficulty in removing the forming needle without damage to the film in any of these applications.

A. S. Baxter.

Cavendish Laboratory,
Cambridge.

1st April, 1951.

[1] *Nature*, **161** (1948), 777.
[2] *Proc. Roy. Soc.* **A** (July 7, 1949).
[3] *Rev. Sci. Instrum.*, **21** (1950), 360.
[4] See for example : W. Kaye, *J. Appl. Phys.* **20** (1950), 1209.

Refrigeration of Oil Diffusion Pump Baffles

Sommaire

ETUDES DE L'EFFET de la réduction de la température du piége—(baffles) d'une jauge à diffusion sure la réduction de la pression de vapeur de l'huile dans un système évacué. Pendant l'expérience le piège, normalement refroidi à l'eau a été réfrigéré à l'alcool, réalisant ainsi une température de -60°C. Details de l'appareil utilisé ont été donnés.

AS IS WELL KNOWN, the usual type of diffusion pump baffle incorporates one or more water-cooled plates or annular rings arranged so that there is no optical path through the baffle chamber. This condition is intended to prevent oil molecules which backstream from the top jet of the diffusion pump from reaching the system under evacuation. However, oil molecules which collide with the baffle plates and with the wall of the baffle chamber will rebound in random directions. In this way, by a series of collisions, a molecule can make its way through the chamber. At each collision, however, it loses a certain amount of

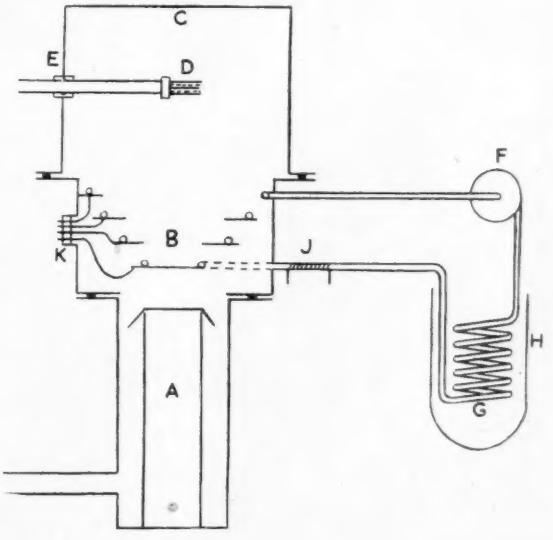

Fig. 1.—A. Diffusion Pump B. Baffle
C. Vacuum Chamber D. Ionisation Gauge
E. Wilson Seal F. Fountain Pump
G. Cooling Coil H. Dewar Vessel
J. Heating Coil K. Kovar Seals.

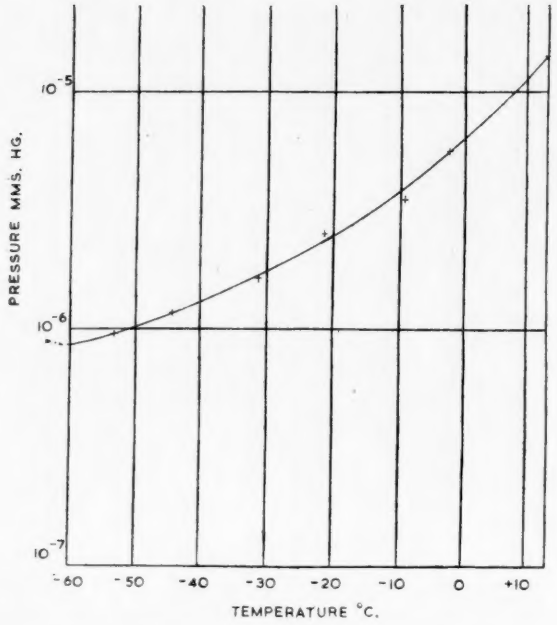

Fig. 2.—Ultimate pressure vs. average baffle temperature.

energy, the loss depending on the temperature of the surface with which it collides and on the accommodation coefficient between the molecule and that surface.

After a number of collisions the energy of the molecule will be reduced almost to a value corresponding to the baffle temperature. Thus the oil pressure above the baffle will be substantially the vapour pressure of the pump fluid at the temperature of the baffle. By lowering this temperature, therefore, one can expect to obtain a lower pressure of oil vapour within the vacuum system. The improvement which can be effected in this way is of considerable importance in many instances, particularly where the vacuum system contains electron emitters which are easily poisoned by traces of oil vapour.

The relation between ultimate pressure and baffle temperature has been investigated using the apparatus illustrated in Fig. 1 (the backing lines and rotary pump are not shown). The six inch diffusion pump A and its baffle B were surmounted by a cylindrical chamber C of 32 litres volume. Pressure measurements were made by means of a tungsten filament ionisation gauge D stripped of its glass envelope and sealed to the glass end of a glass-to-copper seal. The copper end of the seal was soldered to a brass tube which projected into the system through a Wilson seal E. The importance of using an immersion ionisation gauge when making ultimate pressure measurements has been demonstrated by Blears[1].

The baffle B which is normally water cooled (by means of water pipes soldered to the baffle plates) was in this case chilled by alcohol circulated within a closed system by a 'fountain' pump F. Before entering the baffle the alcohol passed through a coil G immersed in a bath H of trichloroethylene and solid carbon dioxide. In this way a baffle temperature of -60°C. was obtainable and could be varied up to +13°C. by means of a heating coil wound round the input pipe at J. The temperatures of the four baffle plates were measured by chromel-alumel thermocouples the leads of which were conveyed through the wall of the baffle chamber by Kovar seals at K.

After pumping the apparatus for 48 hours with the baffle plates at -60°C, readings of ultimate pressure against baffle temperature were taken and are shown graphically in Fig. 2. It should be emphasised that the pressures quoted are *equivalent nitrogen pressures* and not true oil pressures, *i.e.*, gauge readings were converted to pressure by means of a pressure vs. ion current curve obtained by calibrating the gauge on dry nitrogen. The pump fluid was Apiezon 'B' oil.

With the baffle plates at +13°C, the ultimate pressure was 1.5×10^{-5} mm. Hg, which fell to 8.6×10^{-7} mm. Hg at a baffle temperature of -60°C.

Pump speed measurements were made on dry nitrogen with the baffle at room temperature and at -60°C. Plotting speed against partial pressure of nitrogen over the pump (*i.e.* subtracting the ultimate pressure from the total pressure indicated by the ionisation gauge at each observation) there was little to choose between the two curves. At 10^{-3} mm. Hg the speed above the baffle was 190 litres/sec. in each case. At 10^{-4} mm. Hg the speed was 256 litres/sec. with the baffle at room temperature and 245 litres/sec. with the baffle at -60°C. If one plots speed against total pressure, however, then of course the pump speed appears to fall to zero at 1.5×10^{-5} mm. Hg in the room temperature case and at 8.6×10^{-7} mm. Hg in the -60°C. case.

R. S. Barton
A. H. Turnbull

Ministry of Supply,
Atomic Energy Research Establishment,
Harwell, Didcot,
Berks.

6th April, 1951.

[1]Blears, J., *Proc. Roy. Soc.* **A. 188,** (1946), 62.

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BOOK REVIEWS

Kleinste Drucke, ihre Messung und Erzeugung. (Lowest Pressures, Their Production and Measurement) by Dr. RUDOLF JAECKEL. (Springer-Verlag/Berlin, Goettingen, Heidelberg, 1950). Pp. 302. Price £3 9s. 4d.

THE TEMPO OF PROGRESS in vacuum science and technology is daily quickening and although we now have excellent treatises in English on vacuum technique it is increasingly important for the vacuum technologist, in order to keep up-to-date, to keep a careful watch on publications in all languages. It should not be forgotten, particularly in these days when industrial and scientific laboratories, and even homes, abound with numerous vacuum devices, that, at the beginning of this century, rapid pumping equipment did not exist, and that even ten years later the diffusion pump was a thing unknown.

The enormous debt, in this respect, that this generation, and, in particular, present-day science and industry, owes to the genius of the late pioneer in vacuum technique, Wolfgang Gaede, has not yet been properly recognised nor adequately expressed. Gaede invented (1905) the rotary mercury pump, published (1913) the first design of a molecular pump and, last but not least, in 1915 he created the diffusion pump. At the time of his death he was concerned with the development of the 'gas-ballast' pump. An opportunity will be given in a future issue of this journal for a full appreciation of Gaede's work. Dr. Jaeckel, the author of the monograph under review, is the present director of the high vacuum division of Messrs. E. Leybold's & Nachfolger A.G., the firm which not only made the first pumps to Gaede's original designs but which has had the privilege of manufacturing Gaede pumps for nearly fifty years. Therefore, it is only natural that much of Dr. Jaeckel's work reflects the guidance of and his close association with Gaede himself.

The war caused a break between Great Britain and Germany, and a gap in our knowledge of developments in the latter country. Dr. Jaeckel's monograph provides the material with which to fill that gap. Not only does he give an up-to-date detailed and comprehensive survey of the vast range of vacuum apparatus manufactured, and in use, all over the world, but he gives prominence to the various developments, including the 'gas-ballast' pump, which have been made in Germany in recent years.

The contents of Dr. Jaeckel's paper-backed monograph is divided into five sections. As befits a professor of applied physics at the University of Bonn and a director of research, Dr. Jaeckel fully realises the fundamental importance to the vacuum engineer of a good knowledge and understanding of fundamental gas kinetic theory. The first section of the monograph is therefore devoted to a discussion of the basic principles involved in both equilibrium states and in transport phenomena in gases. In the second section the various types and ranges of manometers and pressure gauges are discussed. This follows traditional lines and includes a brief description of leak detectors.

The third section, on pumps, is outstanding for its detail, not only constructional but theoretical. In the discussion of the theory of the vapour-stream pump Dr. Jaeckel adheres closely to Gaede's original ideas. As an introduction he gives a careful thermodynamical analysis of jet phenomena, and then, by suitable simplifications, derives relationships for the speed of exhaustion and the ultimate pressure attainable and their dependence on the geometry of the pump and the physical properties of the gas being pumped. Dr. Jaeckel then refers to a series of experiments, performed by Schroeder and himself with a large oil diffusion pump, in which several of the theoretical relationships were experimentally verified. In the concluding technical part of this section the performances of the various commercial types of vapour pumps using different organic liquids are compared.

The section on outgassing, clean-up by electrical discharge, and sorption of gases by charcoal and metals etc., is contributed by Dr. H. Schwarz and contains much information of practical value. So do the two final sections in which Dr. Jaeckel, with the assistance of Dr. E. Schuller, describes the various forms of vacuum equipment, joints, seals, valves, and auxiliary apparatus including oils, greases and waxes, which are in common use. These sections are perhaps the weakest part of the monograph.

'Kleinste Drucke,' is the ninth in the series of technical physics monographs published by Springer under the general editorship of Professor W. Meissner, is primarily intended to provide the technician engaged in vacuum research and engineering, with the basic facts and the latest practical information. This it does admirably except that it omits all reference

to modern vacuum plant, such as evaporation plant and vacuum stills. It cannot be classed as a textbook. It will be useful as a handy reference book both for the physicist and engineer. The volume contains much hitherto unpublished material. Though the author apologises in the preface for the fact that the references to work published outside Germany during the period 1940-5 may, quite understandably, be incomplete, it is difficult to find that anything of major importance has been overlooked. The detailed list of references (500 in all) to original papers and patent specifications which are attached to each section are extremely valuable and are carefully arranged. The numerous, clear, line diagrams and the vast amount of useful numerical and practical information which is presented in graphical or tabular form will be of great use to those designing vacuum systems for particular purposes. Much time spent in searching through manufacturers catalogues and in computation will be saved by reference in the first instance to Dr. Jaeckel's monograph, and, though it is to be expected that pride of place is given to Leybold's products, readers need not be afraid that they will not find the necessary data relating to the products of firms in Great Britain.

It remains only to say in conclusion that the monograph shows clearly that the tradition established by Gaede is being upheld strongly and in a truly worthy manner.

S. Weintroub

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VACUUM
CLASSIFIED ABSTRACTS

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 —
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 — INSTRUMENTATION
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —
49 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) A Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract *e.g.*, 20/I as distinct from 20/II etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

Cumulative Subject Index

Subject — Abstract No.

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10 — VACUUM ENGINEERING (GENERAL) — 10

Industrial Applications of High Vacuum Techniques 20/I

Spain. A survey is made of the latest developments in vacuum engineering and their application to industrial production. In the electrical field, impregnation of motor windings and condenser paper manufacture is mentioned as well as pumped rectifiers. In the optical field, filters, coating of lenses and production of semi-reflecting mirrors is quoted. Much space is given to the discussion of theoretical and practical aspects of vacuum distillation. Molecular distillation procedures are described as applied in processing vegetable and animal oils, producing vitamins and separating fatty acids. Finally an account is given of the importance of vacuum procedures in the metallurgical field.

Article by E. B. Santiago & L. M. Gonzalez Montes *An. Mec. Elect.*, **26**, Section 3, May-June 1950 131-146

Sommaire : Apercu général des applications de la technologie due vide dans l'industrie. S'étend de l'imprégnation d'éléments électrotechniques à la distillation moléculaire d'acide gras.

Vacuum Metallurgy—A New and Growing Industry 21/I

See Abstract No. : 71/III

Molecular Distillation in Production and in the Laboratory 22/I

See Abstract No. : 80/III

A Method for Measuring the Efficiency of Getters at Low Pressures 23/I

United Kingdom. The quantity of gas, a getter can absorb at a given pressure, is not the only factor which determines the efficiency of a getter material. The amount of free gas left in a vessel after evacuation (*e.g.* in an electronic valve) is usually negligibly small compared with the absorption capacity of the getter used. Thus, the absorption of the free gas is assured, if the getter becomes effective before the free gas settles on other parts of the vessel. This is dependent on the rate, at which the gas is taken up by the getter and to determine this rate for various materials is the object of this investigation. The experimental technique used is based on Knudsen's laws for the flow of gas at low pressures. A small glass bulb of known dimensions is connected to the manifold of a pump by a pumping stem of small diameter. A needle valve for pressure adjustment is fitted to the manifold and an ionization gauge located at either end of the pumping stem to check on the pressure in the glass bulb and manifold respectively. The getter placed in the glass bulb continuously absorbs gas. Thus, the pressure in the bulb is smaller than that in the manifold. Once the conductivity of the pumping stem is established, the volume of gas flowing through the stem and absorbed by the getter per unit of time can be ascertained. Table 1 (reproduced below) gives the gettering rate of a variety of materials examined in this investigation.

Type of Getter	*Rate of gettering in cm³ sec⁻¹* $p_g = 3 \times 10^{-7}$	$p_g = 1.5 \times 10^{-6}$
Mg	—	9
Ba I (bright below mica disk)	—	22
Ba II (bright at centre)	125	135
Ba III (black at centre)	400	260
Th II	1,650	1,500
Zr II	230	190

Article by S. Wagener *Brit. J. Appl. Phys.*, 1, Sept. 1950, 225-231

Sommaire : Description d'une méthode pour mesurer l'efficacité de différentes matières à effet getter aux basses pressions.

Getter Materials for Electron Tubes 24/I

United States. This is a survey article. Getter material is defined as a solid having the property of collecting free gases by adsorption, absorption or occlusion. There are three types of getters :—" Bulk Getters ", metals used in sheet or wire form, specially heated to initiate gettering action and mounted on hot electrodes of the tube for the purpose;—" Coating Getters " generally applied to such electrodes of vacuum tubes which during service are maintained continuously at elevated temperatures in the range of 200—1,200°C.; they usually consist of non-volatile metal powders, sintered upon the electrode surfaces. Finally, " Flash Getters ", chemically active and comparatively volatile metals, usually evaporated at the conclusion of the pumping process. The authors discuss application and performance of getters in general and finally summarize the specific gettering properties of each of nine materials, *i.e.* tantalum, columbium, zirconium, thorium, aluminium, magnesium, barium and phosphorus. Several tables are given, one of which is reproduced below. 74 references.

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Comparison of Getter Efficiency for Metals and Gases

Getter	*Gas*	*Efficiency (in microns of pressure)* *Bright Deposit*	*Diffuse Deposit*
Al	O_2	7.5	38.6
	N_2, H_2, CO_2	—	—
Mg	O_2	20.0	202.0
	CO_2, N_2, H_2	—	—
Th	O_2	7.45	31.15
	H_2	19.45	53.7
U	O_2	10.56	9.26
	H_2	8.9	21.5
Mischmetal	O_2	21.2	50.9
	H_2	46.1	63.9
	N_2	3.18	16.1
	CO_2	2 2	44.8
Ba	O_2	15.2	45.0
	H_2	87.2	73.0
	N_2	9.5	36.1
	CO_2	5.21	59.5

Getter efficiency is determined by the product of volume (1 litre) and pressure of gas cleaned up by 1 mg getter material. Diffuse deposits were obtained by flashing the getter in argon and consist of finely divided getter material.

Sommaire : Résumé des propriétés de neuf matières getter différentes bien connues.

Article by W. Espe, M. Knoll & M. P. Wilder *Electronics*, **23**, Oct. 1950 80-86

The Gettering Efficiency of Thin Barium Films at Low Pressures 25/I

Germany. The investigation reported here aimed at establishing the gettering efficiency of thin barium films in pressure ranges below 10^{-6}mm. Hg. The properties required from gettering materials are discussed and summed up as follows : (*a*) a getter material should not evaporate at the temperature at which the evacuated vessel is being baked, (*b*) it should be effective in adsorbing the common residual gases in the vessel, *i.e.* O_2, N_2, H_2, CO_2 and CO. Finally, (*c*) the vapour pressure of the getter material and that of its reaction products with residual gases should have a negligibly small vapour pressure at the service temperature of the vessel. The experiments conducted for the purpose have confirmed that barium films are still showing a considerable gettering capacity at a pressure level of 10^{-8}mm. Hg. Where such films have been produced by evaporation in a high vacuum they can be used at room temperature, but where they are produced in an argon atmosphere of 5 microns pressure they should be cooled with liquid air.

Sommaire : Etude du comportement de couches mince de Barium avec références spéciales à leurs effets de getter aux très basses pressions.

Article by G. Haase, *Z. Angew. Phys.*, **2**, April 1950, 188-191

On the Possibility of Using the Ionisation of Gas Molecules for the Production of Low Pressures 26/I

See Abstract No. : 45/II

Evacuation Time With Reciprocating Pumps 27/I

See Abstract No. : 44/II

13 — ELECTRICAL SCIENCE — 13

Metallised Paper Capacitors 28/I

United States. A full description is given of production and performance characteristics of metallised paper capacitors as distinct from the foil-type of paper capacitors. The metallisation is carried out *in vacuo* by means of evaporation. The early manufacturing methods used zinc for the metallisation as this is a metal with a comparatively high vapour pressure. In the recent past production has switched to aluminium. Capacitors of this type offer two major advantages. (1) They stand up well to surge voltages due to their self-healing capacity, *i.e.* where sparking occurs under excessive stress, the aluminium coating is caused to melt by the spark discharge and the molten material around the puncture is blown away. As a result a metal-free area is left around the hole producing a high surface resistance in that area affecting only slightly the overall insulation resistance of the capacitor. (2) Metallised paper capacitors are very compact in design and recommend themselves for use wherever space is limited. Typical applications are discussed.

Sommaire : Description de performances et caractéristiques électriques de condensateurs en papier métallisé sous vide.

Article by J. H. Fisher *Electronics* **23**, Oct. 1950 122, 214, 218-222

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Abstract No. and References

Improvements In or Relating To Metallised Paper Capacitors — 29/I

See Abstract No.: 62/III

The Electrical Resistance of Liquid Gallium in the Neighbourhood of Its Melting Point — 30/I

United Kingdom. The electrical resistance of liquid gallium has been measured in the normal and super-cooled state. To prepare a pure sample of gallium, 40 gm. of the metal were melted in a quartz vessel and the oxide converted into the chlorides by addition of a little dilute hydrochloric acid. After maintaining the quartz vessel at red heat for two hours under reduced pressure of 10^{-5}mm. Hg the chlorides boiled off and left pure gallium in the vessel. The gallium was then transferred, still under vacuum, to a resistometer made of pyrex glass (shown in a sketch). Readings were taken for temperatures from 0—50°C. It is shown that the change of resistance in this range is linear. The temperature coefficient of resistance is calculated to be 1.089×10^{-3}°C.; also, the approximate resistivity value is given for 0°C. $= 2.72 \times 10^{-5}$ Ohm. cm.

Sommaire: Méthode pour déterminer la résistance électrique du gallium.

Note by C. Dodd *Proc. Phys. Soc.* **63B**, Sept. 1950 662-664

The Service Life of Glowing Tungsten Filaments and Their Speed of Evaporation in High Vacuum — 31/I

Germany. The conditions affecting the service life of a tungsten filament have been investigated and, in particular, the effect of various temperatures. The service life of a filament comes to an end when the weight reduction caused by evaporation has reached a certain percentage of the original value. This percentage is independent of the actual temperature. Thus, it is possible to calculate the temperature coefficient of the speed of evaporation by measuring the length of service life at different temperatures. Plottings have been made of these calculations for the temperature range of 2,500—2,800°K. The results of this investigation are discussed and compared with work carried out by Langmuir and Zwikker previously. It is mentioned that Zwikker had stated that the service life of a filament comes to an end when its weight is reduced to 15% of its original value. The influence of contaminating gases and vapours is also discussed and it is stated in this connection that the short life of needle cathodes fitted to electron microscopes is not the result of contaminating vapours and gases, but probably an effect of ion bombardment.

Sommaire: Etude des lois régissant la durée de vie de filaments de tungstène sous vide.

Article by E. Bas-Taymaz *Z. Angew. Phys.*, **2**, Sept. 1950 374-376

Method for Treating Filaments of Ionization Gauges — 32/I

See Abstract No.: 58/II

A Universal Power Supply Circuit for Use with Ionization Gauges — 33/I

See Abstract No.: 60/II

Vacuum Tubes with Mutually Bombarding Oxide Cathodes — 34/I

See Abstract No.: 38/IV

Development of the Low Pressure Electrodeless Discharge in a High-Frequency Electric Field — 35/I

United Kingdom. The authors investigated sometime ago the initiation of an electrodeless discharge produced in a cylindrical vessel by a uniform high frequency electric field which is directed parallel to the axis of the vessel. The dimensions of the glass tube are smaller than the mean free path of electrons at the pressure prevailing in the vessel. It was shown then that an electron starting off at one end wall in a suitable phase of the field will initiate an electron avalanche, if, after crossing the tube, it hits the opposite end wall with sufficient velocity. Observations then recorded would lead to the belief that light emitted from the discharge should be brightest at the ends of the tube as the discharge proceeds to develop. However, it is found that the light, in fact, is brightest in the centre and this phenomenon is the subject of the present communication. The authors state that a calculation of the energy of an electron as a function of its position along the tube proves that the electron acquires sufficient energy on its travel through the tube to cause ionization before it reaches the centre of the tube. This applies to all electrons reaching the centre from either direction and this accounts for a considerable build-up of positive space charges in the central area of the tube (to which the applied electrical field is to be added). Eventually new electrons are produced near the centre by the ionization of gas atoms. This is the origin of the electrons which are found in the final stage of the discharge. The equilibrium conditions based on this secondary emission of electrons are described in detail.

Sommaire: Explication du mécanisme de la décharge prenant naissance dans une enceinte dépourvue d'électrodes, placée dans un champ électrique de haute fréquence.

Letter by G. Francis & A. von Engel, *Proc. Phys. Soc.* **63B**, Oct. 1950 823-824

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Abstract No. and References

Some Recent Developments in Rare-Gas Discharge Lamps — 36/I

United Kingdom. The author describes the development of gas discharge lamps beginning with a brief description of the Moore tubes. Much space is given to a description of the latest type, *i.e.* the " gas-arc " lamp. The two main features of this lamp are : a continuous background spectrum extending through the whole visual range and a high efficiency. In this connection details are given of the water-cooled xenon-filled lamp with an input of 5 KW and an efficiency of 30 L/W. The colour of this lamp is the nearest to genuine daylight that could be obtained by an artificial light-source.

Sommaire : Etude des développements récents de la construction de tube à décharge dans les gaz rares.

Article by J. N. Aldington *Endeavour*, 9 Oct. 1950 203-306

Hall Coefficient and Resistivity of Thin Films of Antimony Prepared by Distillation — 37/I

See Abstract No. : 56/III

15 — PHYSICS — 15

Vacuum Differential Thermal Analysis — 38/I

See Abstract No. : 87/III

The Absolute Determination of the Emission Rate of Beta-Rays — 39/I

United States. Hitherto Geiger-Mueller counters have been the principal instruments used for the determination of beta-ray emission rates. The use of these instruments, however, involves the introduction of correction factors for absorption, scattering etc., which it is difficult to assess. When radio-active material emits at least one charged particle per disintegration, a comparatively simple way exists of measuring activity by observing the rate at which the charge is carried away by emitted electrons. This method has been used by a number of research workers but does not yield accurate results partly due to the fact that no means were available then for the provision of an adequate vacuum. A method is described here which is based on the procedure mentioned but yields more accurate results. Measurements are effected in a large tank 42 inches in diameter and 46 inches in height which can be exhausted to a pressure as low as 10^{-6}mm. Hg. At this pressure level ionization effects, which may affect the accuracy of the results, are completely eliminated. The actual rates of emission were determined by the Townsend method. Data is given for two isotope materials, P^{32} and Na^{22}. Summing up, it is stated that the experimental equipment described can record much lower energies than equipment based on the use of counters.

Sommaire : Description d'une méthode pour déterminer l'émission aux rayons béta qui serait un progrès par rapport à la méthode du tube Geiger-Mueller.

Article by R. K. Clark *Rev. Sci. Instrum* 21, Aug. 1950 753-759

Motion Picture Studies and Electron Bombardment of Colloidal Crystals — 40/I

See Abstract No. : 65/II

Evaporation of Zinc and Zinc Oxides in Electron Bombardment — 41/I

United States. Thermionic emission, secondary electron and photo-electric emission are very sensitive to the atomic and molecular constitution of the surface in question. The mass spectrometer would be an ideal tool for the examination of surface constitutions if means could be found of removing atoms and molecules from the surface of a solid for identification in the mass spectrometer. The investigation reported here was initiated to establish whether electrons of themselves could cause the dissociation of atoms from a surface or whether such dissociation phenomena (reported by Baldock and others previously) are due exclusively to the heating produced by the electrons. A Dempster mass spectrometer using a 180° deflection was employed in these experiments. Two electron beams, parallel to each other, were placed immediately in front of the mass spectrometer slit, the one nearest to the slit was acting as the ionising beam and the other was used for the bombardment of the target. The target support consisted of a tantalum strip which could be heated and the temperature measured with a thermocouple. The beam striking the target could be varied in its intensity whereas the ionising beam had a constant intensity. The materials to be investigated were deposited by evaporation *in vacuo* on the tantalum strip. Two series of experiments were carried out with each material, (1) heating the tungsten strip without electron bombardment and (2) heating the tungsten strip whilst under bombardment. Results are given for zinc and zinc oxides. The rate of evaporation of zinc did not appear to be affected by electron bombardment, but zinc oxide showed an appreciable change. The behaviour of the latter cannot, in the view of the author, be explained as a purely thermal effect and this leads to a discussion on the interaction of dissociation and de-excitation forces in atoms of the surface which are energised by the electron beam.

Sommaire : Etude des possibilités de prélèvement des atomes et molécules d'une surface par leur identification au spectromètre de masse et de la possibilité qu'offre la méthode pour l'étude des surfaces de solides.

Article by F. E. Dart *Phys. Rev.*, 78 15.6.1950 761-764

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Abstract No. and References

Mass Spectrometric Study of Solids. I. Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials — 42/I

See Abstract No. : 35/IV

The Action of a Gas on a Solid — 43/I

See Abstract No. : 28/IV

The Ultimate Limits of Sensitivity of Lead Sulphide and Telluride Photo-Conductive Detectors. — 44/I

United Kingdom. Photo-conductive layers of lead sulphide and telluride can now be produced with sensitivity properties in the near infra-red region of the spectrum many times greater than those of the conventional radiation detectors, for instance, bolometers and thermopiles. Coupled with rapid response times such layers can be very useful as detectors in spectroscopy, etc. The author investigates the theoretical limits of the sensitivity of such cells proceeding on the assumption that the limit is imposed by fluctuations in the radiation quanta reaching the layer. The results of the calculations are very largely dependent on the different spectral sensitivity curves. Also, it was established that cooling the layer worsens the limiting sensitivity. In the case of the PbS cell, the detection limit can be improved appreciably by cooling the cell surroundings.

Sommaire : Examen des limites théoriques de cellules photo-conductive à base de sels de plomb en fonction de la différence de leurs courbes de sensibilité spectrale et en variant la température de la couche sensible.

Article by T. S. Moss *J. Opt. Soc. Amer.* 40, Sept. 1950 603-607

A Method of Determining Concurrently the Thickness and Refractive Index of a Thin Film or Lamina — 45/I

United Kingdom. An optical method is described for the determination of the thickness of thin films which does not require a knowledge of the refractive index. A Michelson interferometer is used and set so that the optical path difference between the two beams is constant everywhere. The light issued from the interferometer enters the slit of a spectrometer, the mirrors and the slit being at conjugate distances from the collecting lens. The film, the thickness of which is to be determined, is introduced into the longer optical path, and this alters the relationship between the order m and the wave number of the dark bands. The orders m_1, m_2, and m_3 of each dark band are determined and plotted against their wave numbers. Formulae are given to calculate the thickness from these values and subsequently the refractive index.

Sommaire : Une méthode optique par la mesure de l'épaisseur de couches minces ne nécessitant pas la connaissance de l'indice de refraction.

Letter by P. D. Fochs *J. Opt. Soc. Amer.* 40, Sept. 195 623

The Thickness Measurement of Thin Films by Multiple-Beam Interferometry — 46/I

See Abstract No. : 51/III

Reflectivity of Thin Silver Films and Their Use in Interferometry — 47/I

See Abstract No. : 55/III

Calculation of Interference Filters — 48/I

France. Calculations are made for interference filters consisting of multiple layers. Reflectivity and transmission coefficients are calculated for normal incidence by means of recurrence formulae. The calculations are given for transparent quarter-wave films and for half-wave films. The formulae establish that the selectivity depends on the number of films. (*Science Abstracts*)

Sommaire : Calcul de coëfficient de reflexion et de transmission pour filtres interférentiels à couches multiples.

Article by J. Roig & C. Descamps *C. R. Acad. Sci. Paris*, 230 27. 2. 1950 827-829

16 — CHEMISTRY — 16

Antibiotics — 49/I

United States. The development of antibiotics is surveyed and data given on the respective production facilities available today. Besides penicillin, the following antibiotics have been developed in the last twelve years : Streptomycin, aureomycin and terramycin. Last year 58 plants were in operation in various parts of the world producing penicillin at a rate of 32 million million Oxford units.

Sommaire : Aperçu historique du développement des antibiotiques et tablau des capacités de productions actuelles dans différents pays.

Article by J. R. Skeen *Chem. Engng.* 57 Sept. 1950 265-267

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I — General Science and Engineering — I
Contd.

Abstract No. and References

Changes in the Reactivity of Purified Prothrombin After Freeze-Drying 50/I

See Abstract No. : 33/IV

Direct Determination of Oxygen in Compounds Containing Carbon, Hydrogen and Oxygen 51/I

United States. The technique described here is based on the Unterzaucher carbon-reduction procedure. It can readily be used for organic substances containing carbon, hydrogen and oxygen but it can probably be extended to the simultaneous determination of hydrogen and nitrogen as well as oxygen. Its accuracy is claimed to be within 1% and its application to cover analyses over the whole range of oxygen content from 0.1 to 100%. The procedure consists essentially of pyrolyzing the sample in a stream of helium and circulating the decomposition products in a closed system over carbon at 1100°C. Hydrogen is removed through a heated palladium membrane and by observing the thermal conductivity changes of the gas mixture, the rate of formation of carbon monoxide is followed. After completion of the conversion the total gas volume is measured and the carbon monoxide content determined by calculation. Solid, liquid and gaseous samples can be used. Details are given of the equipment employed. For outgassing pressures as low as 5×10^{-5}mm. Hg are required. The actual analysis is carried out at a pressure level of 500—600 mm. Hg. A single oxygen determination takes approximately one hour and samples up to three grams a piece can be analysed by this method.

Article by S. Siggia J. G. Hanna & I. R. Kervenski *Analyt. Chem.* **22**, Oct. 1950 1295-1304

Sommaire : Description d'une méthode par la détermination de l'oxigène dans des corps organiques contenant du carbone, de l'hydrogène et de l'oxigène, basée sur le procédé " Unterzaucher " de réduction par le carbone.

An Investigation of High Vacuum Freeèe Drying as a Means of Drug Preservation. Part I 52/I

See Abstract No. : 32/IV

The Concentration of Tocopherols from Natural Sources by Molecular Distillation 53/I

See Abstract No. : 81/III

19 — SCIENCES (other than the above) — 19

Infiltrated or Impregnated Alloys 54/I

United Kingdom. This is a survey article. The practice of infiltration or impregnation has been established for quite some time in specialised branches of industry, *e.g.* in the manufacture of self-lubricating bearings. However, it's importance, in particular, to powder metallurgy has only partially been realised so far. Due to porosity early products of powder metallurgy were lacking in physical properties compared with cast or wrought articles. For instance, the tensile strength of 0.3% carbon steel powder, pressed and sintered, could be improved three times its original value by infiltration with copper. Work carried out in U.S.A., Austria and Germany is reviewed and a list quoted which was prepared by Kieffer and Benesowsky enumerating the essential conditions for the successful application of infiltration techniques. This list mentions the benefit derived from vacuum procedures in this connection, stating that such procedures are particularly advantageous, where the infiltrating metal does not tend to wet the sintered material.

Article by H. W. Greenwood *Engineer* **190**, 20.10.50 384-385

Sommaire : Discussion de l'importance des méthodes d'imprégnation métallique sous vide ou autrement, par rapport à la métallurgie des poudres en général.

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Evacuation Time with Reciprocating Pumps — 44/II

United States. This note refers to an article by Hicks published previously (see abstract No. 1/II). The method of calculating pump-down times suggested in that article is criticised as being too rough. Rigorous calculation, it is said, is simpler and more accurate. A formula is given for use in such calculations and its accuracy proved by way of an example.

Sommaire : Données de la formule permettant de calculer exactement la durée d'évacuation de pompes alternatives pour des cas bien spécifiés.

Note by E. F. Johnson Jr. *Chem. Engng.* **57**, Sept. 1950 125

On the Possibility of Using the Ionisation of Gas Molecules for the Production of Low Pressures — 45/II

France. It is well known that an electric discharge between two electrodes in a rare-gas atmosphere causes a reduction of pressure in the vessel and this phenomenon is often used industrially, *e.g.* to improve the vacuum of electronic valves. It can be assumed that this effect is closely linked with the ionisation process. Probably, the gaseous ions are hitting the electrode with such force that they are absorbed at the same time. To examine the potentialities of such an " Ionisation Pump " an experiment was conducted which is reported here. A constriction was made in the centre of a glass tube and an electrode placed on either side of the constriction. An ionisation gauge was connected to the centre of the constriction. After pumping down to 10^{-1} mm. Hg a voltage of approximately 2,000 was applied to the electrodes. A discharge developed with an initial current of 15 mA. After 10 seconds the pressure in the constriction had fallen to 10^{-4} mm. Hg and the value of the discharge current to 5 mA. When the discharge was interrupted the pressure in the constriction was building up again to its original level in a very short time. Re-starting the discharge, the pressure was reduced in the same way as before. Interpreting the results of the experiment, the author advances the theory that molecules coming from the gauge are ionised on entering the discharge path and proceed to the respective electrode, where they are neutralised, subsequently travelling on into the space beyond the electrode. It is obvious that the phenomenon will only occur as long as there is sufficient carrier gas to maintain a discharge.

Sommaire : Description d'une expérience prouvant que la décharge électrique entre deux électrodes dans une athmosphère de gaz rare entraîne l'abaissement de la pression dans l'enceinte.

Article by R. Champeix *C.R. Acad. Sci. Paris*, **231** July 1950 40-42 see also *Le Vide*, 5 Nov. 1950 912-913

Performance of Vapour-Lift Pumps under Vacuum — 46/II

United Kingdom. The authors were faced with the problem of transferring the bottom of one vacuum still to the feeding point of another. In large scale operations one can use centrifugal pumps for this purpose but this is not economical in the case of small scale operations considered here. In addition it was considered objectionable in this case to use rotary positive displacement pumps and, therefore, an experimental vapour-lift pump was built which was operated under a pressure of 2—10 inches Hg absolute. Theory and operation of this pump were fully explored and are recorded in this article. A. o., it was found that a vapour-lift pump operates satisfactorily under a vacuum as low as 2 inches Hg absolute. Further, the rate of lift increases with the relative submergence and possibly also with a reduction in the net height lifted. All tests were carried out with dipentene as a test liquid. Finally, an installation is described of two vapour-lift pumps in a commercial distillation plant.

Sommaire : Rapport des travaux expérimentaux en vue du développement de pompes à transfert de vapeur, utilisées pour le transfert de la nappe de fond d'un alambic à vide à un autre.

Article by N. R. Mukherjee & H. R. C. Pratt *Industr. Engng. Chem.*, **42** Sept. 1950 1883-1894

The Characteristics of Diffusion Pumps — 47/II

United Kingdom. This paper is intended as a contribution to the search for an effective method of comparing diffusion pump performances of different design. It deals at length with the " intrinsic " and " operational speed " of pumps, terms introduced in a previous publication, the former being an attribute of the pump itself, the latter a function of the system. " Efficiency " is interpreted as the ratio of the intrinsic speed to the speed of the perfect pump. A series of experiments is described aimed at showing the dependence of these qualities on heat input, backing pressure, jet design and slot dimensions. Finally, Gaede's statement is discussed that the optimum speed of a diffusion pump is obtained when the mean free path of the gas molecules in the cloud of back streaming vapour molecules is equal to the width of the gap. More research work is called for, a. o., on the problem, whether it is correct to assume that " diffusion " conditions apply at low heater wattages and " ejector " conditions at high heater inputs. The effects of such assumptions on jet and gap width design, are discussed.

Sommaire : Comparaison des caractéristiques de pompes à diffusion en vue de trouver un dénomminateur commun permettant de comparer équitablement les performances de pompes de différentes origines.

Article by R. Witty *Brit. J. Appl. Phys.*, 1 Sept. 1950 232-237

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II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

On the Vapour Flow in the Diffusion Pump

48/II

Japan. Little is known on the theory of vapour flow in a diffusion pump. One investigation has been published by Molthan re. the effects on the pumping speed caused by vapour flowing back into the vacuum system. The author criticises Molthan's findings claiming that Molthan oversimplified matters by assuming, for instance, spherical symmetry of velocity distribution. The author makes a fresh approach to the subject by treating the conditions at the nozzle hydrodynamically and the region of highly rarefied vapour by applying the kinetic theory of gases. In this manner he finds the degree, by which the intensity of the back flow is affected by the pressure of the vapour, by its Mach number and by the width of the clearance connecting the working section of the pump with the vessel to be evacuated. The author comes to the conclusion that supersonic velocity of the vapour flow has a favourable effect on the pumping speed.

Sommaire : Etude théorique des conditions d'écoulement de vapeur d'une pompe à diffusion en vue d'établir les facteurs favorisant une vitesse de pompage optimum.

Article by S. Oyama *J. Phys. Soc. Japan*, **5**, May-June 1950 192-197

High Speed Mercury Diffusion Pump

49/II

United States. It is proposed to improve the speed of the conventional type of mercury pumps by providing pump chimneys with an effective cross sectional area of such dimensions as to deliver the mercury vapour to the pumping jets in a pre-determined ratio. As additional means of control, the pump chimneys are dimensioned so that each of them extends for a determined distance into the next larger chimney, thus creating an impedance path for the vapour. Actual figures are suggested in this patent for the ratios to be applied and for the proportional dimensions of the respective chimneys.

Sommaire : Proposition de dimensions de tuyeaux de pompe à diffusion qui augmenteraient la vitesse de pompage.

C. R. Stoltenberg *U.S. Pat.* 2,499,839

Measurement of the Pumping Speed of Rotary Pumps

50/II

France. The author describes methods and apparatus employed for the measurement of pumping speed. Results are given of actual tests covering a variety of rotary pumps of French and American make.

Sommaire : Description de méthodes et d'appareils pour la mesure de la vitesse de pompes rotatives.

Article by R. Henry *Le Vide*, **5** July-Sept. 1950 859-865

Pumped Rectifiers in 600-Volt Transportation Service

51/II

United States. Details are given of pumped rectifiers of the latest design used in transportation power supply systems. The units consist essentially of a high voltage a.c. switch gear and transformer section, a pumped ignition rectifier, a d.c. switch section and auxilliary control apparatus. Particulars are given of each of these components, but the vacuum equipment is not described in great detail. All protective devices included in the equipment are also described.

Sommaire : Détails de construction d'un redresseur du type à pompage continuel.

Article by R. Siegel *Gen. Elect. Rev.* **53**, June 1950 28-31

21 — GAUGES — 21

Shop Facilities for Testing Pressure Instruments—Part I

52/II

United States. Details are given of equipment recommended for checking and calibrating pressure indicating instruments of the vacuum or high-pressure type. In this connection a test station for vacuum gauges is described and diagrammatically illustrated. It consists basically of an air supply and bleed valve system. Observations are taken with a lock-in vacuum. A liquid-column manometer pressure standard is used which can be evacuated at both ends by two independent rotary pumps. Finally, details are given of the piping system and the intricate arrangement of the mechanical valves required for the plant.

Sommaire : Description d'un banc d'essai pour le contrôle et le calibrage de jauges à vide et à pression.

Article by W. D. Wood *Taylor Technol.* **3**, Autumn 1950 24-25

A New Method of Measuring Pressure Electrically

53/II

United States. In connection with the development of a magnetic oxygen analyser, a pressure measuring unit was designed to cater for the correction of errors introduced by changes in absolute gas pressure of the sample. Essentially it consists of a sealed chamber containing argon gas of a known pressure which can be heated (electrically) until the pressure inside the chamber exceeds the pressure of the gas sample to be measured on the opposite side of the diaphragm which forms one wall of the chamber. This diaphragm carries one of two contacts of an electrical circuit which incorporates a thermistor as the temperature-sensitive element. In the case just mentioned the contacts will separate and turn off the heat. When the chamber cools down, the contacts will close again. Eventually the chamber temperature will stabilise at a value proportional to the pressure being measured. In fact, this device is measuring the pressure of the sample gas by means of resistance thermometry. The accuracy of the instrument is claimed to be 1 mm. at 740 mm. Hg.

Sommaire : Description d'un dispositif indicateur de pression basé sur l'application du thermomètre à résistance.

Article by E. C. Blom *Instruments* **23**, Sept. 1950 903

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

A New Vacuum Gauge

54/II

United States. A new type of vacuum gauge is described which can be operated at all levels of sub-atmospheric pressure, *i.e.* in all ranges ordinarily covered by three types of gauges complimentary to each other. It works on the principle that an a.c. signal can be obtained from a normally d.c. pressure gauge by cyclically changing the pressure in a container at a given frequency. The gauge consists essentially of two bellows driven by the eccentric shaft of a small motor and hereby changing their volumes by about 20% during each cycle. Resistance wires are fitted to the inside of each bellows as detector elements and each bellows is connected to the vacuum system by an appropriate aperture. The pressure in the bellows is changed almost every 0.02 second. Due to the small size of the hole most of the gas which has penetrated into the bellows cannot escape in that short period of time. Thus the greater part of the percentage change in volume is realised as a percentage pressure change and, when the pressure surrounding the wire is actually changed by the motion of the bellows, a periodical pulsating signal is developed, the magnitude of which corresponds to the average pressure obtaining in the bellows. In the pressure range above 50 mm. Hg the resistance wire no longer acts as a Pirani gauge but merely records the thermal energy changes of the gas. As an alternative to wires, a platinum ribbon was used by the designers and its performance is shown in a graph. In place of resistance wires a thermocouple or an ionisation gauge can be incorporated in this gauge.

Sommaire : Nouveau principe de jauge, facilitant la mesure de pression à tous les niveaux entre 1 atm. et 10^{-8} mm. Hg.

Article by R. Havens, R. Koll & H. LaGow
Rev. Sci. Instrum. **21**, July 1950 596-598

Diaphragm Micromanometer with Electrical Pick-Up

55/II

United States. The instrument described has been developed for the measurement of differential pressure in the micron region by the National Bureau of Standards (see NBS Technical Report No. 1475). It has been designed for use in connection with mass spectrometers. Existing instruments are liable to give inaccurate readings mainly for the reason that the sample is measured prior to its being passed into the reservoir of the mass spectrometer where it expands to a thousand times its original volume. Its pressure is then assumed to be 1/1,000 of its original value but this assumption does not take into account losses due to adsorption by the stopcock grease and walls of the system. The new instrument is intended for use in the actual reservoir of the mass spectrometer. It consists of a pressure cell and an electrical micrometer enclosed in a glass jar which can be evacuated or filled to any desired reference pressure. The cell consists of a thin corrugated diaphragm, sealed at the periphery to a slightly dished brass disc and is connected to the gas sample so that a change of the pressure of the gas causes the diaphragm to move. The movements of the diaphragm are translated electrically and are indicated by a mutual-inductance micrometer. The latter is placed over, but not in contact with, the diaphragm. The instrument covers the pressure range of 1-100 microns and has a sensitivity of 0.1 micron on a 50 micron scale.

Sommaire : Détail de construction d'un micromètre à diaphragme développé spécialement pour l'utilisation avec des spectromètres de masse.

Note by Anon.
Instruments, **23** Nov. 1950 pp. 1136

A Philips-Type Ionisation Gauge for Measuring of Vacuum from 10^{-7} to 10^{-1} mm. Hg

56/II

United States. A cold cathode ionisation gauge of the Philips type is described covering the extended range of pressures shown in the title. The gauge is operated from a 2,000 volt d.c. power supply, and employs a magnetic field of 2,000 gauss. The anode consists of a nichrome wire V loop, the cathode consists of stainless steel discs. The whole design is exceptionally small and compact, and due to the use of a specially balanced amplifier its sensitivity is about six times higher than that of the common hot-filament ionisation gauge. Calibration is linear at low pressures and non-linear in the micron range.

Sommaire : Rapport sur une construction améliorée de la jauge Philips englobant la gamme de pression de 10^{-1} à 10^{-7} mm. Hg.

Article by E. C. Evans & K. E. Burmaster
Proc. Inst. Radio Engrs., N. Y., **38** June 1950 651-654

Extension of the Low Pressure Range of the Ionization Gauge

57/II

United States. Details are given of an improved ionization gauge. The design described here is based on the results of investigations into residual current phenomena as observed in ordinary ionization gauges at a pressure level of 10^{-8} mm. Hg. The residual current at this pressure level is due to soft x-rays which are created at the grid by the incidence of the thermionic electrons. The x-rays release photo-electrons from the ion collector. The main feature of the new design is the re-arrangement of the 3 basic elements of the gauge as a result of which the ion collector intercepts only a small fraction of the x-rays produced at the grid. It is claimed that the new gauge gives reliable pressure readings down to 10^{-9} mm. Hg.

Sommaire : Discussion de la construction améliorée de la jauge à ionisation permettant la mesure de pression jusqu'à 10^{-9} mm. Hg.

Article by R T. Bayard & D. Alpert
Rev. Sci. Instrum. **21**, June 1950 571-572

Method for Treating Filaments of Ionization Gauges

58/II

United States. A process of treating metallic filaments intended for use as electron emitters in ionization gauges which comprises treating the filament with a liquid hydrocarbon and subsequently heating it in an

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Contd.

Abstract No. and References

evacuated envelope in the presence of a minute amount of hydrocarbon adhering to the filament as a result of the prior treatment, thereby reducing the gas sensitivity of the filament.

Sommaire : Il est proposé un traîtement à faire subir aux filaments métalliques à utiliser dans les jauges à ionisation qui réduirait leur sensibilité en fonction des gaz présents.

H. W. Washburn
H. Wiley &
Consolidated
Engng. Corp.
U.S. Pat.
2,406,419

The Service Life of a Glowing Tungsten Filament and Its Speed of Evaporation in High Vacuum

See Abstract No. : 31/I.

59/II

A Universal Power Supply Circuit for Use with Ionization Gauges

60/II

Holland. The uses of ionization gauges are briefly discussed. The necessary requirements which the power supply and indicating circuits have to meet are defined, and a circuit satisfying these requirements is fully described. Particular attention is paid to the design of the current generator and stabilizer, micro-ammeter circuit and automatic cut-out circuit. (*Science Abstracts*)

Sommaire : Détails d'un circuit d'alimentation universel pour jauges à ionisation.

Article by
J. W. A. van der
Scheer
P T T-Bedrijf,
3 May, 1950
24-29

22 — MEASURING PLANT (VACUUM) — 22

Application of Electron Diffraction to the Study of Surface Films

61/II

France. The uses of electron diffraction methods in connection with research work on the structure of surface films less than 10A thick are discussed. Actual applications of the method are described, *e.g.* investigation work carried out on oxide films which have formed on zinc and aluminium.

Sommaire : Discussion et démonstration de l'emploi de la diffraction électronique par l'étude de couches minces sur la base de procédés expérimentaux pour les recherches sur les couches minces d'oxyde.

Article by
M. H. Raether
Rech. Aeron.
May-June 1950
45-49

An Apparatus for the Study of Insulating Surfaces by Means of Electron Diffraction

62/II

France. The examination of the surfaces of insulating material in an electron diffraction camera proves difficult because the electron beam directed to the target at a glancing angle shows a tendency of being deflected and diffused. This is the result of static charges produced in the material. The use of a beam of ' slow ' electrons has been suggested as a remedy but is not really satisfactory. The neutralisation effect of slow electrons depends on a number of factors, three of which are : the intensity of the beam, the electrical conductivity of the specimen and its thickness. In addition, there is the disadvantage that the neutralisation effect does not last long enough to cover prolonged examinations. The authors investigated these effects and found that they are due to tungsten evaporating from the electron gun and depositing on the target in the form of an amorphous layer. A reduction of the tungsten filament temperature is no solution as this will reduce the supply of electrons. But changing from tungsten to platinum activated by barium and strontium oxides permits the reduction of the filament temperature to about 700°C. A new type of electron gun is proposed, design and operation of which is explained in detail It is claimed that the steadiness and intensity of the diffraction picture does not change even in cases where the examination takes longer than an hour, if the new type of electron gun is used.

Sommaire : L'examen de surface d'isolents par la diffraction électronique a présenté des difficultés. Il est proposé une solution pour les vaincre.

Article by
J. J. Trillat &
S. Oketani
J. Rech. Centr. Nat. Rech. Sci.
No. 12, 1950
148-149
see also
Le Vide,
5 July-Sept. 1950
827-830

Apparatus for the Study by Electron Diffraction of the Effect of Temperature on Surface Films

63/II

United Kingdom. The structural changes of layers of long-chain carbon compounds on metal surfaces produced by variation of temperature have been examined in an electron diffraction camera and wide variations were reported in the temperature of transition from an oriented to a disordered structure. To make the temperature measurement of the specimens as accurate as possible, holders were specially designed for heating specimens to be examined ' by reflection ' and for heating ' transmission ' specimens. Finally, a description is given of a new type of roll-film holder.

Sommaire : Description de porte-objets et d'un support pour film en bobine, en partant des procédés d'examen au diffractographe électronique.

Note by
J. W. Menter &
J. V. Sanders
J. Sci. Instrum.
27, Dec. 1950
335-336

The Field Emission Electron Microscope Makes Molecules Visible

64/II

Germany. It has recently been reported that images of single molecules (phthalocyanine molecules in the case reported), have been obtained in a field emission microscope. The molecule in question is fifty times smaller than the smallest so far made visible in a standard electron microscope. The instrument consists essentially

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of a tungsten wire with a fine tip sealed into an evacuated bulb which is coated on the inside with a phosphorous layer. In operation, cold emission (also called : field emission) of electrons takes place from the tip of the wire and these electrons reach the phosphorous layer on the inner bulb surface producing a glow where they impinge. The material under examination is evaporated on to the wire tip, increasing locally the emission of electrons and thus creating a brighter spot on the phosphorous layer. A magnification of the order of one million is theoretically possible with an instrument of this type. Thus, it may be possible to collect evidence hitherto unobtainable on the migration and deposition of large molecules upon surfaces. However, the design of the instrument is not finalised yet and needs further development, particularly, regarding the vacuum equipment required.

Sommaire : Détails d'un microscope électronique à émission froide plus simple que les types standard.

Article by Anon. *Discovery*, 11, Sept. 50 274-5

Motion Picture Studies of Electron Bombardment of Colloidal Crystals — 65/II

United States. The behaviour of colloidal crystals under electron bombardment was observed in the electron microscope and motion picture records taken. In the case of a sodium chloride crystal subjected to electron bombardment the pictures show water being driven off and sections of the crystal becoming transparent to electrons. Another series of pictures shows the same crystal as it changes toward the residual envelope condition which is its final state. The origin of the residual envelope is discussed at length and it is stated that it has been observed that salts which are water-soluble tend to leave residual envelopes, whilst insoluble salts do not. The residual envelope is a replica of the original crystal and provides information on the surface of the original crystal. The microscope used gave a magnification of 7,200 and the pictures were taken at a rate of about 16 frames per second. The value of the motion picture technique is particularly pronounced in the early stages of the bombardment as a number of transient phenomena follow each other in very rapid succession during that period.

Sommaire : Résultats de l'étude " Cinématographique " du comportement des cristaux collodîaux soumis au bombardement électronique.

Article by J. H. L. Watson & L. E. Preuss *J. Appl. Phys.* 21, Sept. 1950 904-907

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Evaporation of Zinc and Zinc Oxides in Electron Bombardment — 66/II

See Abstract No. : 41/I

Mass Spectrometric Study of Solids. I. Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials — 67/II

See Abstract No. : 35/IV

Mass Spectrometric Analysis of Low Concentrations of Vapours in Air — 68/II

See Abstract No. : 26/IV

R.F. Field Mass Spectrometer — 69/II

United States. Details are given of a three-stage non-magnetic mass spectrometer employing the principle of velocity selection. The instrument has been developed by the National Bureau of Standards, and is suitable, a. o., for the determination of surface reactions separately analysing positively and negatively charged components. The instrument uses neither bending nor focussing. The ions produced in the ionization chamber travel on parallel paths through a glass cylinder 8 inches long and 2 inches wide. Three sets of three tungsten wire grids are spaced along the tube to form three stages. The r.f. potential is applied to the middle grid in each stage. An additional grid with a negative charge turns back any electrons which may have arisen anywhere along the tube. The collector plate at the end of the tube-is charged with a potential, sufficiently positive to repel all but the desired positive ions.

Sommaire : Détails d'un spectromètre de masse à 3 étages utilisant le principe de la sélection des vélocités.

Article by Anon. *Electronics* 23, June 1950 176-182 see also *Chem. Age* 63, 19.8.50 259-260

Diaphragm Micromanometer with Electrical Pick-Up — 70/II

See Abstract No. : 55/II

26 — MATERIALS — 26

Vacuum Tests of Rubber, Lead and Teflon Gaskets and Vinyl Acetate Joints — 71/II

United States. Rubber, lead, teflon and vinyl acetate have been tested for gas evolution in a vacuum. Such gas evolution can either be due to the evaporation of part of the material itself or, alternatively, to the liberation

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of gases occluded in the material. The former are ' condensable ' gases in all cases, the latter not necessarily so and an attempt was made in the course of the experiments described in this article to determine the amount of condensable gases present. The experimental equipment consisted of a vacuum chamber of approximately half a litre volume which could be exhausted by means of a mercury pump at a speed of 20 litres per second. An ionization gauge was used for the pressure readings. A valve was provided to shut off the chamber from the vacuum pump. Gaskets of the various materials to be tested were used to seal the vacuum chamber and after pumping down the valve was closed and the pressure recorded as a function of time. These pressure-time curves for the initial rate of gas evolution and recordings of the subsequent equilibrium pressure with the valve open constitute the basis for a comparison of the properties of the material studied. The results of these experiments are shown in tabulated form and an extract of the table is given below :

Flange Sealing Material	*Total Pumping Time (hr.)*	*Equilibrium Pressure (mm. Hg) when pumping on chamber and in equilibrium with*		*Apparent Vapour Pressure (mm. Hg)*			*Approximate Initial Rate of Gas Evolution at 20°C.*
		Dry Ice	*Liquid Air*	20°C.	—78°C.	—180°C.	*(mm. liters/sec.)*
Buna S rubber gaskets (Greene No. 5900)	70	1×10^{-5}	5×10^{-6}				0.7×10^{-6}
O-ring rubber gaskets	40	1×10^{-5}	5×10^{-6}				2.5×10^{-6}
Lead Gaskets	40	1×10^{-6}	3×10^{-7}	none	none	none	10^{-9}
Vinyl acetate	2	5×10^{-6}				2×10^{-6}	1.5×10^{-7}
(chamber at 20°C.)	100	2×10^{-5}		2.5×10^{-6}		1×10^{-6}	3×10^{-9}
	300 at 20°	4×10^{-7}	3×10^{-7}	5×10^{-7}		4×10^{-7}	10^{-9}

Sommaire : Essai du comportement de caoutchouc, plomb, teflon et acetate de vinyle sous vide.

Article by R. W. Cloud & S. F. Philp *Rev. Sci. Instrum.* **21**, Aug. 1950 731-733

Apparatus for Determining Gas Permeability of Carbon and Graphite 72/II

See Abstract No. : 80/II

The Electrical Resistance of Liquid Gallium in the Neighbourhood of Its Melting Point 73/II

See Abstract No. : 30/I

28 — APPARATUS COMPONENTS — 28

Packless Valve Flow Regulator 74/II

United States. The valve described can be used at pressures above or below atmospheric and is designed for the control of very low flow rates which make it imperative to avoid valve stem leakage, a common risk in conventional designs. Essentially the apparatus consists of a chamber filled with porous material fabricated from fritted glass, unglazed porcelain, sintered metal or the like. There is an entry port to the chamber for the fluid and an exit port. A hole is drilled through the porous material of the chamber connecting the entry port and a mercury reservoir. By means of a piston the mercury in the reservoir can be made to rise through the hole to any height in the chamber. The control of the flow rate, therefore, depends on the maintenance of a constant pressure drop across the valve as well as on the mercury plunger position determining the resistance to flow offered by the valve. The design facilitates the calibration of piston position versus flow rate for given conditions of fluid viscosities and fluid pressures. Flow rates as low as 10^{-3} cc./sec. can be regulated, employing a particular grade of porcelain as the porous element.

Sommaire : Principe et performance d'une vanne sans presse-étoupe développée pour la régulation de très faibles débits.

Article by W. Rose *Rev. Sci. Instrum.* **21**, Aug. 1950 772-773

An Effective Vacuum Cut-Off 75/II

United States. A cut-off, made of glass and quartz, is described and shown in a sketch which is still efficient in a vacuum of 10^{-5} mm. Hg against atmospheric pressure. Its essential parts are a circular plate made of quartz 1 mm. thick and 25 mm. diameter placed on the polished end of an 18 mm. wide pyrex tube. This plate is connected inside the tube by means of a quartz fibre to a quartz encased iron armature. The upward force required to seat the disc firmly on the tube is provided by a quartz cantilever spring from which the assembly is suspended. This upward force of the spring is counter-balanced by the downward force of a solenoid on the armature when the cut-off is to be opened.

Sommaire : Un clapet en verre et quartz est décrit et appliqué sur un schéma diagrammatique.

Note by G. W. Sears & E. R. Hopke *Rev. Sci. Instrum.* **21**, June 1950 570

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Glass Variable Microleaks for Gases

76/II

United States. Details are given of a variable microleak for application in spectroscopy and positive ray work. It consists essentially of an inner and outer tube joined by a ring seal and a spot seal at the end of the inner tube. Gas leaks from one tube into the other through a crack in the inner tube. The opening through the crack can be varied continuously by twisting the free ends of the tubes slightly. Details are given, how to produce the crack. The following are the maximum and minimum rates of air for a series of such leaks with a differential pressure of 1 atmosphere. The values in cc./hr. n.t.p. are : 9.6-0.012; 6.3-0.015; 4.2=0; 60-0.01; 4.8-0.01; 120-0.15; 40-0.03; 60=.0; 4.3-0.2; 40-0.01; 3.6-0.006; 6-0.003.

Sommaire : Description de micro-fuites en verre avec indi cation de leur débit d'emploi.

Note by J. J. Hopfield *Rev. Sci. Instrum.* **21**, July 1950 671-2

29 — MISCELLANEOUS APPARATUS — 29

Improvements In or Relating to Absolute Pressure Controlling Apparatus

77/II

United Kingdom. Improved accuracy of performance is claimed for the apparatus described in this patent. The apparatus consists of a closed chamber connected to the system which is subjected to the pressure control by the apparatus. A valve is provided inside the chamber which is operated by bellows. The bellows can be adjusted in such a manner that the throttling action of the valve can be initiated at any predetermined pressure level.

Sommaire : Détails de construction d'un regulateur depression qui donnerait des résultats plus précis que les modeles existants.

Wallace & Tieranan Products Inc. *Brit. Pat.* 646,784

Leak Testing Device

78/II

United States. If an evacuated system is being tested for leaks, a change of pressure indicated on the gauge is not necessarily an indication of the presence of a leak. It is the object of this invention to provide for an arrangement whereby the tracing of a leak can be made more foolproof than in the past. It is proposed to provide two gauges, one of the ionic type and one of the non-ionic type, for reading the pressure of the system under test and to scan the system with a gas which has ionization characteristics different from those of air. In operation a discrepancy between the pressure readings of the two gauges will clearly indicate the presence of a leak, whereas identical changes of pressure readings on both instruments will indicate a change of conditions in the system not connected with the presence of leaks.

Sommaire : Description d'un dispositif de recherche de fuites permettant le montage en ligne de jauges ionique et non-ionique, pour augmenter la facilité d'identifier l'origine de fluctuations de pression mesurées.

D. W. Sherwood & U.S. Atomic Energy Comm. *U.S. Pat.* 2,507,321

Measuring Low Vapour Pressures

79/II

Italy. An apparatus for measuring vapour pressures between 10^{-6} and 10^{-2} mm. Hg at temperatures between —50°C. and +150°C. is described. The main feature of the method is the use of a lead or wire vacuum gauge of the Knudsen type, the readings of which are independent of the gas used in the above pressure range. It is shown how to avoid all uncertainties due to gases occluded in the material under examination and gas evolution from the apparatus. The necessary operations take a few hours, outgassing included.

Sommaire : Détails d'un appareil mesurant des pressions de vapeur entre 10^{-6} et 10^{-2}mm. Hg à des températures entre —50°C. et +150°C.

Article by I. Filosofo M. Merlin & A. Rostagni *Il Nuovo Cimento* **7**, 20.1.1950 69-75

Apparatus for Determining Gas Permeability of Carbon and Graphite

80/II

United States. An apparatus is described which is simple and cheap to produce claimed to give an accuracy of measurement of 0.3%. Results can be expressed either in comparative or absolute units. It essentially consists of two standard borosilicate glass flasks of 500 and 250 cc. volume respectively, forming two compartments of the apparatus with the test specimen in between them. A third compartment, a 50 cc. bulb of the same material, is sealed to the bottom compartment and serves for the admission of a measured amount of gas. Two mercury manometers are used, one connected to each of the major compartments. Before use the apparatus is evacuated (by means of a mechanical pump) to a pressure of about 100 microns and left sealed for some time to make sure that it is air-tight and no evolution of gases occur. Then gas is admitted into the small bulb and from there into the lower compartment. The pressure of the gas in the two lower compartments is read on the (lower) Hg-manometer and by means of a stop watch the time is measured until a predetermined pressure value is indicated on the Hg-manometer of the upper bulb, indicating the rate of permeation. Finally, details are given of the calculation required to determine the quantity of gas that passes through the sample in a given period of time.

Sommaire : Description d'un appareil simple fait de pièces standard pour la détermination de la perméabilité aux gaz du carbone et du graphite.

Article by S. D. Fulkerson & F. J. Lambert *Analyt. Chem.* **22**, Oct. 1950 1310-1312

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II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

A Joule-Thomson Cascade Liquefier for Helium

81/II

United Kingdom. Details are given of a new helium liquefier installed recently at the Royal Society Mond Laboratory, Cambridge. Its main features are: a closed hydrogen circuit—the liquid hydrogen produced is never decanted from the machine—and a reduced starting time for the production of liquid helium. After some 30 minutes liquid hydrogen is produced and after some 40 minutes liquid helium. Liquid nitrogen is consumed at a rate of 8 kilograms per hour and liquid helium is produced at a rate of 3.8 litres per hour. The machine can also be used as a hydrogen liquefier giving an output of 5 litres per hour.

Sommaire: Détails d'un liquéfacteur d'hélium comprenant un nombre de dispositifs nouveaux installé récemment au Royal Society Mond Laboratory à Cambridge.

Article by J. Ashmead *Proc. Phys. Soc.* **63 B**, 1.7.50 504-508

Vacuum Crystallizers

82/II

United States. A brief description is given of (batch) vacuum and (continuous) vacuum crystallizers in an article on crystallizers in general. Advantages offered by the use of vacuum plant: Low operating costs, relatively easy control and regulation of the process, temperatures attainable below that of the available cooling water, and, finally, absence of heat transmission through a solid medium that may become scaled, salted up or corroded. In the case of the batch crystallizer the charge is introduced hot, in the form of a highly concentrated solution, and is subjected to a gradually increasing vacuum which effects cooling and evaporation at the same time. At the desired low temperature level the vacuum is broken and the charge drained off for filtering, etc. In the case of continuous production feed and drainage is continuous. Cooling below temperature of the cooling water can be effected by the use of a steam-jet thermo-compressor. Non-condensable vapours are removed by a two-stage stem-jet air ejector with intercondenser.

Sommaire: Cristallisateurs à vide pour fonctionnement par paquets ou continuel mentionnés et brièvement décrits.

Article by A. R. Thompson *Chem. Engng.* **57**, Oct. 1950 125-132

A System for the Collection and Purification of Radon by Remote Control

83/II

United States. A description is given of a radon collection and purification system in use at the K.25 Laboratories. The system is heavily shielded and operated exclusively by remote control. 230 mg. of radium are held in the plant. The unit is very similar in its design features to the one described by Livingstone previously. However, all stopcocks incorporated in the previous design are replaced by mercury cut-offs. Details are given of the operation of the plant and it is stated that only 30 minutes are required for collection, not counting the initial and final pumping operations. This represents a considerable gain on time compared with the Livingstone plant fitted with stopcocks.

Sommaire: Description d'un système collecteur et purificateur de Radon. Dispositif principal: Vannes à mercure en place de robinets.

Article by C. H. Shiflett M. E. Steidlitz & W. Davis *Rev. Sci. Instrum.* **21**, Oct. 1950 842-844

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

Calculation of Interference Filters 49/III

See Abstract No. : 48/I

Method of Determining Concurrently the Thickness and Refractive Index of a Thin Film or Lamina 50/III

See Abstract No. : 45/I

The Thickness Measurement of Thin Films by Multiple-Beam Interferometry 51/III

United States. Thickness measurements were carried out with gold, copper, aluminium, antimony and chromium films. The thickness of the films examined ranged from 40Å to 1,500Å. Details are given of the equipment used including a diagrammatic sketch. Instead of monochromatic light, white light and a spectroscope were employed. This facilitated the use of microscope slides as substrates in place of optical flats and thus all measurements could be made simply from the wave-length drum of the spectroscope. Details are also given of the method used for the preparation of the films to be measured. The authors state that silver is the ideal material for the reflective coating required and refer to Avery's statement that chromium tends to produce inaccurate results. Where materials other than silver are used for the top coating it is advocated to provide an intermediate layer of the same material as that of the film to be measured. Microscope slides are comparatively thin and make it possible to obtain very low interference orders. The advantages of low order fringes in connection with this experimental technique are discussed.

Sommaire : Détails d'un procédé de mesure de l'épaisseur de couches minces par interférométrie à rayons multiples.

Article by G. D. Scott T. A. Mclauchlan & R. S. Sennett *J. Appl. Phys.* **21**, Sept. 1950 843-846

Evaporation of Thin Films of Uniform and Controlled Thickness. Their Application to Electronics 52/III

France. The various types of electronic valves make use of practically all the specific physical and chemical properties of solid thin films. In the case of television and radar tubes, one often has to apply films accurate in uniformity and thickness. The author describes methods used in production to obtain coatings (by evaporation *in vacuo*) of a uniformity of approximately 1% over an area of 20 cm. in diameter. Methods of controlling the thickness of the deposit within 1/50 of a micron during the actual evaporation process are also described.

Sommaire : Description des méthodes utilisées dans la production de tubes électroniques pour contrôler l'uniformité et l'épaisseur des films déposés.

Article by C. Dufour *Le Vide*, 5 July-Sept. 50 837-841

The Ultimate Limits of Sensitivity of Lead Sulphide and Telluride Photo-Conductive Detectors 53/III

See Abstract No. : 44/I

The Photo-Absorption of a Thin Film of Barium 54/II

See Abstract No. : 37/IV

Reflectivity of Thin Silver Films and Their Use in Interferometry 55/III

United Kingdom. The resolving power of instruments based on the interference of multiple-beams is largely dependent on the reflective properties of the semi-transparent silver films used. In the region of high reflectivities ($R > 0.75$), no accurate data for reflection and transmission are available. It is the object of this investigation not only to establish values of the reflectivity as a function of transmission and wavelength but also to establish the scatter of reflection values for films produced under similar conditions and the changes of reflective properties due to ageing. 30 samples were prepared by evaporation *in vacuo*. In each evaporation three sets of two glass plates were used and placed at 18, 20, 22 cm. distance respectively from the evaporation source in the chamber. Each plate was measured four times : immediately after deposition and 3, 8 and 22 days after processing. The measurements were made with a spectrometer using a photomultiplier as the detecting element. Discussing the results the authors state that coatings of approximately equal density appear to vary in their reflectivity values according to their position in the vacuum chamber during evaporation. Plates positioned close to the evaporation source seem to have higher reflectivity values than plates positioned further away. Finally the measurements described show that—certainly for wavelengths of more than 5,000Å —a considerable increase in resolving power could be achieved, if etalon plates with more perfect surfaces were available.

Sommaire : Etude des propriétés de réflexion de films d'argent utilisés en interférométrie.

Article by H. Kuhn & B. A. Wilson *Proc. Phys. Soc.* **63B**, Oct. 1950 745-755

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III — Vacuum Processing Techniques — III
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Abstract No. and References

Hall Coefficient and Resistivity of Thin Films of Antimony Prepared by Distillation

56/III

Canada. An a.c. method is described for measuring the Hall coefficient. Antimony layers of exceptional purity were required for these experiments. To reduce probabilities of contamination during the evaporation process and difficulties in the outgassing procedure, an all glass apparatus was constructed in which to carry out the evaporation process. The necessary heat was supplied from outside by a 700 watt furnace which could be lowered over the apparatus. 600°C. was required for the deposition of the antimony layers. This temperature level is sufficiently low for the apparatus to be made from pyrex glass. At temperatures above 600°C. the apparatus should be made from quartz. Very good results were achieved with the apparatus. The only impurities present were those contained in the metal itself and it was found that such substances with a boiling point lower than that of the pure metal tended to evaporate more rapidly producing the effect of a fractional distillation.

Sommaire : Description d'un appareil utilisé pour le dépôt d'antimoine par évaporation. Il contient des dispositifs facilitant une pureté exceptionnelle du dépôt.

Article by W. F. Leverton & A. J. Dekker *Phys. Rev.*, **80** 15.11.1950 732-736

The Dielectric Strength of Aluminium Oxide Films

57/III

United Kingdom. Under the influence of an electric field, electrons in a crystal will gain energy from the field and at the same time lose energy to the lattice vibrations. Electrical breakdown will occur, if electrons already possessing an energy greater than the ionization energy gain additional energy from the field. If the mean free path of the electrons is decreased by introducing foreign ions into the crystal, or by raising the temperature, the dielectric strength will increase. In a thin film of a dielectric the dielectric strength will rise as the thickness decreases. The author investigated whether this theory, promoted by Froehlich in 1937, applies also to films of amorphous materials in a thickness range of 100Å or less. Accordingly, aluminium oxide films prepared by partially anodizing thick aluminium films deposited by evaporation on optical flats were investigated. Details are given of the method, by which the breakdown current was measured. Throughout the experiments the films were kept in a vacuum and at a temperature of 15°C. Detailed results are shown in graphic form. As the thickness increased from 100Å to 1,000Å the dielectric strength decreased from 11×10^6 V/cm. to 6×10^6 V/cm.

Sommaire : Examen de films d'aluminium d'une épaisseur d'environ 100 Å et 1.000 Å respectivement, concernant leur valeur diélectrique. L'on trouve que la qualité diélectrique diminue en fonction inverse de l' épaisseur.

Letter by P. D. Lomer *Proc. Phys. Soc.* **63B**, Oct. 1950 818-820

The Adsorption of Gases on Copper Films

58/III

United Kingdom. In an endeavour to throw light on the mechanism underlying low-temperature oxidation (the subject of theories advanced previously by Mott and Cabrera), a study was made of the adsorption and subsequent reaction of oxygen with a number of copper films prepared in varied temperature conditions. An all-glass apparatus was used consisting of a sphere with a tungsten filament at its centre, the latter to serve as the evaporation source for the copper. The bulb was sealed to a manifold containing the oxygen supply and a Pirani gauge. At the start of the experiment the sphere was evacuated and the copper outgassed. Subsequently the sphere was sealed off and the copper evaporated onto the walls of the bulb. After completion of the evaporation the seal between the bulb and the manifold was broken and oxygen admitted to the desired pressure. Finally the system was shut off from the pump and subsequently all measurements were taken. A series of three experiments were carried out covering three different temperature conditions ranging from +74°C to —183°C. The results are given in tabulated form. The table shows that the adsorptive properties of the films at low temperature and their subsequent behaviour at room temperature are strongly dependent on the thermal treatment to which the films have been subjected. In addition there are marked differences in appearance of films of comparable weight per unit area prepared under different conditions. Films deposited at room temperature have the normal copper red appearance, those deposited at —183°C. are bronze in colour, and if annealed at room temperature, the colour changes to the normal copper red. On the other hand, films saturated with oxygen at —183°C. and subsequently warmed to room temperature evolve practically no oxygen and their appearance does not change.

Sommaire : Recherches sur l'adsorption de l'oxigène par des films de cuivre et les réactions en dérivant.

Article by J. A. Allen & J. W. Mitchell *Discuss. Faraday Soc.*, No. 8, 1950 309-314

Determination of Heats of Adsorption Using Metal Films

59/III

United States. Heats of adsorption have an important bearing on the mechanism of hetrogeneous analysis and their determination offers a means of measuring the inter-action of catalyst and adsorbate. The apparatus used in the experiments consists essentially of a thin glass tube, the bottom part of which is fitted with a platinum wire coil. This tube is sealed into an outer tube forming a vacuum-tight jacket and provided with an inlet for evacuation or, alternatively, the admission of cooling water. The platinum wire coil serves as the detector element of a resistance thermometer. Details of the circuit are given. The deflection of a galvanometer, included in this circuit, serves to ascertain the heats of adsorption with the help of a formula given. In a typical experiment the apparatus is evacuated and outgassed at 500°C. After cooling down the metal film is deposited by evaporation from a hair-pin shaped wire *in vacuo*. Subsequently, the apparatus is immersed in a thermostat and hydrogen admitted in small increments. The results of the experiment seemed to indicate that

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the initial heats of adsorption on a sparsely covered surface increase from rhodium to tungsten and all heats decrease with the fraction of surface covered. It has further been found that the heats of adsorption at room temperature and —183°C. are identical and that absorption does not take place at room temperature until 75% of the surface is covered. Sixteen different films were investigated, half of them orientated and half unorientated. Graphs are shown recording in detail adsorption phenomena on iron and nickel films.

Sommaire : Etude de seize films métalliques différents en vue de déterminer les chaleurs d'adsorption. L'on utilisa l'hydrogène dans les expériences.

Article by O. Beeck W. A. Cole & A. Wheeler *Discuss. Faraday Soc.*, No. 8 1950, 314-321

The Gettering Efficiency of Thin Barium Films at Low Pressures — 60/III

See Abstract No. : 25/I

Application of Electron Diffraction to the Study of Surface Films — 61/III

See Abstract No. : 61/II

Improvement In or Relating To Metallised Paper Capacitors — 62/III

United Kingdom. The use of metallised paper as distinct from plain paper and metal foil has assisted successfully in reducing the size required of capacitors for a given capacity value. However, in the manufacture of very small capacitors difficulties are still encountered in making rolls of metallised paper with few turns of a sufficiently small diameter, rigid enough to be handled without deformation. To overcome these difficulties it is proposed to use a single strip of metallised paper and to separate the metallisation on the strip by a de-metallised band extending in a devious course along the strip and forming a repeated pattern. Thus, the metallisation on the strip is divided into two separate areas. In assembly, winding the strip into a roll, the two metallised areas of the roll overlap each other. The metallised area extending from one edge is connected to one pole of the capacitor and the metallised area extending from the other edge is connected to the other pole.

Sommaire : Description d'un procédé de fabrication permettant la production de condensateurs de dimensions exceptionnellement réduites, utilisant le principe de la métallisation.

A. H. Hunt Ltd. & R. A. Grouse *Brit. Pat.* 647,573

Metallised Paper Capacitors — 63/III

See Abstract No. : 28/I

How to Metallize Glass and Other Ceramics — 64/III

United States. A summary is given of the methods of applying metal coatings to glass and ceramics. Spraying is discussed and chemical reduction processes as well as electro-plating and electro-forming techniques. Finally, vacuum evaporation and sputtering methods are mentioned and the advantages they offer to this type of production.

Sommaire : Sommaire des méthodes de métallisation du verre et des céramiques.

Article by J. Sturr *Ceramic Industr.* Sept. 1950 78-78 & 105

Two Improved Methods of Graticule Production Using Evaporated Metal Films — 65/III

Australia. Various processes of producing graticules are described. One method well known for its accuracy requires the coating of a glass blank with a wax resist, then ruling it and finally etching it with hydrofluoric acid. The present investigation aimed at the elimination of the etching process connected with that method. As a result a new method was developed which provides for the application of two coatings to the glass, an undercoat of zinc and a top coat of chromium. Both layers are deposited by evaporation *in vacuo* to ensure good adhesion. After deposition of the zinc coating the plate is ruled and after the application of the top coating the plate is immersed in diluted sodium hydroxide solution which causes both coatings to flake off except where the chromium is in direct contact with the glass. Graticules produced in this manner do not cause distortion in optical systems, but have the disadvantage that they cannot be repaired by refilling the lines.

Sommaire : Description de méthodes pour la production de réticules par l'évaporation sous vide pour le dépôt métallique, éliminant ainsi l'attaque de l'acide fluorhydrique.

Article by P. M. Aitchison & R. E. Aitchison *Aust. J. Appl. Phys.*, 1 March 1950 75-79

31 — CATHODIC SPUTTERING — 31

The Practice of Cathodic Sputtering — 66/III

France. It is stated that the theory of cathode sputtering is still not finalised. There are two hypotheses : one theory interprets the emission of metal from the cathode as a simple thermal evaporation process caused by

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

the high temperature of the cathode layer. The other assumes that such emission is caused by an energy transfer from the gaseous ions to the metal molecules. The author deals essentially with the practical side of cathodic sputtering and gives details of the cathode, anode and other plant components required. He states that the optimum pressure in a sputtering chamber is 0.1 to 0.02 mm. Hg dependent on the gas atmosphere, in which the process is carried out. A table is given setting out the gas atmospheres recommended in connection with the sputtering of various metals.

Gas	*Speed of Sputtering in Decreasing Order*
Air	Pd, Au, Ag, Pt, Cu, Cd, Ni, Fe, Al, Mg.
Nitrogen	Ag, Au, Pt, Pd, Cu, Ni, Fe.
Argon	Cd, Ag, Pb, Au, Cu, Pt, Ni, Fe, Tu, Zn, Si, Al, Mg.
Hydrogen	Ag, Au, Pb, Zn, Cu, Fe, Ni, Tu, Co, Mo, Mn, Cd, Al, Cr, Ta, Mg.
Oxygen	Zn, Ag, Au, Pb, Cu, Fe, Mo, Tu, Ni, Cd.

Article by M. E. H. Bacquet *Le Vide*, 5 Nov. 1950 916-917

Sommaire : Court exposé des méthodes de projection cathodique avec quelques conseils pratiques utiles aux opérateurs de tels équipements.

33 — IMPREGNATION — 33

A Swedish Method of Wood Preservation

67/III

Sweden. Wood preservation was rather ineffective until vacuum and pressure methods of impregnation were introduced. Two such methods are mentioned : the ' full-cell ' and the ' empty-cell ' method. The introduction of these methods solved a great number of difficulties experienced in processing but no really effective chemical agent was available. The chemical formula is given of an arsenical compound which has recently been developed and is claimed to be superior in its effectiveness over materials used in the past, reducing at the same time the cost of the impregnation process.

Article by J. Grindrod *Chem. & Industr.* 23.9.1950 668

Sommaire : Bref exposé de procédés d'imprégnation de bois mentionnant un nouveau produit chimique récemment développé en Suède.

36 — DRYING — 36

Double Distillation

68/III

United States. The Eastern Regional Research Laboratories of the Department of Agriculture developed a process of making frozen apple juice concentrate which, on reconstitution with water, tastes like the natural product. The apple juice is evaporated at atmospheric pressure until about 10% of its original volume have been vaporised. This latter volume contains the volatile flavour of the natural product and is processed into an essence by fractionation, reducing it to 10% of the volume evaporated in the first instance. The bulk of the juice, reduced to 90% of its volume in the initial evaporation, is further evaporated under vacuum to about 15% of the original bulk and then the essence just mentioned is admixed. Whilst this method was immediately successful in the case of apple juice, the procedure had to be modified to process grape juice with equal success. 25% of the original bulk was evaporated in the first phase and used for distillation in a fractionating column to produce one gallon of essence for every hundred gallons of juice. 25% of this essence was re-evaporated to give a second even more highly concentrated essence which was admixed to the bulk of the juice after the latter had been further evaporated to a density of approximately 70° brix under a vacuum of 28 inches Hg. and at a temperature of 100°F. 100 gallons of juice concentrated in the manner described yield approximately 18 gallons of fully flavoured concentrate.

Article by Anon. *Chem. Industr.* 67, Oct. 1950 560

Sommaire : Discussion d'une méthode de concentration de jus de fruits récemment développée par le Eastern Regional Researche Laboratories du Département d'Agriculture des Etats Unis, ayant pour but de retenir entièrement le goût du produit naturel.

Apparatus and Method of Evaporation and Concentration of Liquids

69/III

United States. This patent refers to the processing of food products such as fruit juices and the like. To prevent the deterioration of the product during processing and in order to minimize the loss of essential flavour producing ingredients it is proposed to speed up the process of concentration by passing the liquid quickly through a single-pass evaporator under a high vacuum, fractionally condensing the vapours produced in the evaporator and separating the condensable flavour-containing liquid in concentrated form. The latter is collected in a container into which the bulk concentrate is fed after pasteurization whilst still at a temperature within pasteurization range.

G. G. Zahm & Hurd Corp. *U.S. Pat.* 2,512,513

Sommaire : Une méthode est décrite qui a pour but de faire des concentrations de jus de fruits avec un minimum de perte de composés parfumants.

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Contd.

Abstract No. and References

New Evaporator Robotized for High Output Efficiency — 70/III

United States. Details are given of a new plant for the production of concentrated citrus juices,essentially consisting of a high-vacuum low-temperature three-stage thermally-compressed evaporator completely automatically controlled. The evaporator has a vapour removal capacity of 14,500 lbs. per hour, operating at a temperature of 70°C. and concentrates the juice in three steps of 18° brix, 29° brix and 55° brix respectively. The total output of the two units described is 1,174 gallons per hour. Details of the processing sequence are given and of the automatic controls in use. In particular, a full description is given of a newly developed density controller, the instrument controlling the concentration of the final juice in °brix within ±0.1°.

Sommaire : Description d'un nouvel évaporateur utilisè pour la production de jus de citron concentré, à fonctionnement automatique.

Article by C. D. Atkins F. W. Wenzel E. Fehlberg & L. E. Slater *Food. Industr.* **22**, Sept. 1950 58-60

37 — METALLURGICAL PROCESSES — 37

Vacuum Metallurgy—A New and Growing Industry — 71/III

United States. This is an article reviewing the application of vacuum techniques in the metallurgical field. After a short introduction describing the essential features of pumps and measuring instruments an account is given of vacuum techniques applied to the reduction of magnesium, calcium, sodium, lithium and barium. Further, details are given of the purification of metals *in vacuo*. Finally, vacuum distillation as applied to metallurgy is described a.o. the commercial perfection of the Isbell process for the de-zincing of lead. The article closes with a reference to zirconium production and the vacuum casting of beryllium compounds.

Sommaire : Revue des applications de la technique du vide à la métallurgie des métaux.

Article by A. W. Schlechten *Engng. Min. J.* **151** July, 1950 71-73

The Accurate Determination of Carbon in Low-Carbon Steels — 72/III

United Kingdom. Due to the development of stainless and silicon steels with a progressively lower carbon content, the problem of accuracy in carbon determination is becoming more and more serious. The author mentions Yensen's low-pressure combustion method as the first method known to be successful. Ziegler, Wooton and Guldner and others improved it but all versions of the method quoted have the disadvantage that complete absorption of carbon dioxide in barium hydroxide solution is difficult to achieve. As a result of an investigation of these methods an apparatus was built which provided for the isolation of the carbon dioxide in a liquid oxygen trap with subsequent measurement of its pressure after the gas had been transferred to a bulb of a known volume, but it was not any longer considered necessary to carry out the actual combustion under reduced pressure. The low-pressure measuring equipment introduced by Yensen at the time was modified by providing a mercury vapour pump and a Toepler pump to transfer the carbon dioxide from the liquid oxygen trap to the calibrated measuring bulb. Details of the apparatus and the procedure are given. It is claimed that the apparatus is appreciably simpler than previous designs and its accuracy of the same order. The combination of one combustion unit with two liquid oxygen traps facilitates individual determinations at a rate of one every 20 minutes.

Sommaire : Méthode d'analyse du carbone dans les aciers à faible teneur de carbone, laquelle est un développement et une amélioration de la méthode dite de combustion sous faible pression de Yensen.

Article by J. E. Wells *J. Iron & Steel Inst.*, **166** Oct. 1950 113-117

Surface Hardening of Ferrous Metals — 73/III

United States. A method is described of producing a hardened surface on ferrous metals. The surface of the ferrous metal is heated to about 700°C. and is then brought into contact with gaseous diborane. Metal boride is formed as a result. The process is carried out in a chamber kept at a pressure of 30 mm. Hg. At higher pressures non-volatile boron hydrides are formed which will accumulate on cooled surfaces in the apparatus and diminish the yield of hardened metal from a given amount of gaseous diborane. The boride layer deposited on the article is about 5 microns thick and claimed to be extremely hard and abrasion-resistant.

Sommaire : Processus de trempe superficielle de métaux ferreux par dépôt de couches de borures.

H. Schlesinger G. W. Schaeffer & U.S. Atomic En. Comm. *U.S. Pat.* 2,494,267

Infiltrated or Impregnated Alloys — 74/III

See Abstract No. : 54/I

Copper-Titanium Coating and Bonding Process — 75/III

United States. The process protected by this patent deals with the application of a comparatively thick layer of copper-titanium to the surface of a metal article containing copper as a substantial component and is a development of the procedure covered by U.S. Patent 2,351,798 which deals with the application of thin layers of copper-titanium to the surfaces of similar articles. The new process provides for the use of a mixture of metallic copper and titanium hydride in a very finally divided state. This mixture is applied to the article to be coated

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preferably in the form of a paste. After the application of the paste the article is heated up in a muffle to a temperature higher than 900°C. but below that of the melting of copper, and kept at this temperature for about 30 minutes. During that period elementary titanium is formed and combined with the finally powdered copper present. Thus a low melting alloy of copper and titanium is produced which will flow over the whole surface of the article to be coated. During the heating period and the subsequent cooling period a vacuum or an inert gas atmosphere is maintained in the muffle. Such a coating of copper and titanium alloys forms an intimate bond with the copper content of the article itself.

Sommaire : Ce brevet décrit une méthode de recouvrement d'articles en cuivre par une couche de titanium cuivreuz.

P. P. Alexander & Metal Hydrides Inc.
U.S. Pat. 2,512,455

Improvements in the Manufacture of Metallic Titanium

76/III

United Kingdom. A method is described which aims at improving the procedure protected in the (Kroll) U.S. Patent 2,505,854. This latter patent provides for the manufacture of metallic titanium by the reduction of titanium-tetrachloride with molten magnesium at a temperature of 750°C. in a vacuum or, alternatively, an inert atmosphere. It has been found that this procedure has a number of disadvantages. For instance, after cooling down, it is difficult to remove the solidified contents from the crucible. Also, the titanium metal is liable to oxidise during the period, the magnesium chloride is being dissolved. It is proposed, therefore, to modify the procedure and to separate a large part of the magnesium chloride from the titanium, whilst the reduction is still in progress and the magnesium chloride still in a liquid state. A special furnace fixture is required to facilitate the separation in the liquid state, details of which are given.

Sommaire : Ce brevet a trait à la fabrication de titanium métallique par réduction du tetra-chlorure de titanium. Il est proposé un procédé qui serait une amélioration de celui précédemment publié par Kroll.

L. Saunders & British Aluminium Co. Ltd.
Brit. Pat. 638,840

Zirconium

77/III

United Kingdom. The production of zirconium is discussed and a short historical introduction given on the development of the methods in use today. The iodide process initiated by van Arkel and de Boer and the Kroll process are described. Finally an account is given of the properties of commercially available zirconium. The author expects that zirconium will take the place of tantalum in many applications and points out the importance of zirconium as a structural material for atomic power plants.

Sommaire : Discussion de la méthode du Iodure et de celle de Kroll pour la fabrication de zirconium et exposé des propriétés du zirconium du commerce.

Article by G. L. Miller
Industr. Chem. Oct. 1950 435-442

A Laboratory Arc-Furnace for Melting Alloys Containing Refractory Transition Metals

78/III

United Kingdom. A short survey is given of arc-furnaces developed in the past for the preparation of refractory materials such as tungsten, titanium, etc. Difficulties are still experienced in using such furnaces and processing such materials. They are largely due to the high temperature required and the inert materials needed for the construction of the melting chamber. A new design is described in this article which is claimed to overcome these difficulties. One main feature of the new design provides for the specimen to rest on a water-cooled copper hearth which at the same time is the positive pole of the d.c. arc. An inert atmosphere of pure argon protects the charge. Tungsten, in batches of 50 gm. at a time, can be melted in a few minutes, alloys of uranium or titanium are obtained untarnished and zirconium charges remain malleable after melting. The vacuum is produced by a rotary pump and the figure of 20-25 cm. Hg is quoted as a suitable pressure (after the admission of argon). The whole furnace is assembled on a frame which carries also the vacuum pump, valves, gauges and the argon cylinder.

Sommaire : Détails d'un four à l'arc de laboratoire pour la préparation de matières réfractaires telles que tungstène et titanium.

Article by G. A. Geach & D. Summers-Smith
Metallurgia, 42 Aug. 1950 153-156

An Horizontal High Vacuum Electrical Resistance Furnace

79/III

Canada. Prior to designing the furnace described in this article an investigation was made into the reasons why the development of vacuum furnaces and their introduction to industrial practice is comparatively slow. A. o., it was found that (*a*) it is considered expensive to buy and operate such equipment and (*b*) difficulties are experienced due to the presence of leaks in the vacuum system, the methods of their detection and their elimination. The authors make a point of stating that it may not be generally known that vacuum engineering is sufficiently far advanced today to provide for solutions meeting effectively both these objections. However, the evolution of gases from refractories used in the furnace still presents a serious problem. The furnace design described here is intended to reduce the difficulties experienced in this respect, by cutting down to a minimum the amount of refractory material used in its construction. The furnace is of cylindrical shape and its outer walls are made of tinned cast iron. The muffle is made from zircon. Neoprene gaskets are used to allow for low pressures. One oil diffusion pump is provided having a speed of 275 litres per sec. backed by a rotary pump of 5 litres per sec. 4 kw are required to maintain a temperature of approximately 1300°. The oven has been used

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for three years and details of its operational record are given. In the view of the authors, this furnace, originally developed for use in the laboratory only, could be developed successfully into a furnace of the production type more readily acceptable to industry than previous designs.

Sommaire : Exposé des raisons pour lesquelles l'introduction dans la pratique industrielle des fours à vide est tenté. Il est donné des détails d'un tel four, lequel pallierait à certaines des difficultés rencontrées préalablement.

Article by W. E. Kuhn & O. W. Ellis *Canad. J. Res.* **28**, Jan. 1950 1-20

38 — DISTILLATION — 38

Molecular Distillation in Production and in the Laboratory

80/III

Germany. This is a historical survey of the development of molecular distillation processes and their application in the field of organic chemistry. The use of elimination maxima in the place of boiling point determinations is described and demonstrated in connection with the distillation of vitamin A and D from cod liver oil.

Sommaire : Exposé sur l'application de la distillation moléculaire à la préparation industrielle et les travaux de recherche de laboratoire.

Article by L. W. Masch *Chem. Ing. Tech.* **22**, 14.4.50 141-146

The Concentration of Tocopherols from Natural Sources by Molecular Distillation

81/III

United Kingdom. A molecular still is described which is largely based on designs suggested by Hickman. The still is used for the preparation of mixed tocopherols from five varieties of seed oils. The still is of the falling-film type. An oil diffusion pump, a fore pump and suitable traps are provided. The distilland is degassed and fed into the distillation chamber. The distillate is collected in a gutter at the bottom of the condensing cylinder and passed by gravity through a valve into a small chamber with a receiver bottle attached. The valve is closed and a tap opened to let air into the chamber without breaking the vacuum in the main part of the apparatus. This facilitates an exchange of receiver bottles without interrupting the operation of the still. The results of the experimental run are given in tabulated form.

Sommaire : Description d'un appareil à distiller de laboratoire pour la préparation de tocophérols mélanges en partant d'huiles de graines.

Paper by J. Green & P. R. Watt *J. Sci. Food Agric.* May 1950 157-162

Guided Free-Film Distillation Apparatus

82/III

United States. The apparatus described here is of the short-path distillation type and intended for use in connection with lubricating oils, waxes, oil containing soaps and organic mixtures of therapeutically active materials. Its main object is to provide an effective means of preventing the distilland from coming into contact with the surface in the chamber supplying the heat required for evaporation. Such surfaces naturally have a temperature higher than that required for the evaporation of the material and may decompose the material on contact. In the past the provision of spraying devices was advocated to pass the distilland in front of the heat source but it is difficult to confine such sprays to ' safe ' areas. It is proposed, therefore, in the patent described here to provide a number of rods in a vertical position in the distillation chamber and a top plate with a number of apertures at a determined distance from the rods so that the distilland flowing through the holes forms a film between the rods during its passage through the chamber.

Sommaire : Description d'un appareil à distiller pour le traitement d'huiles de graissage, cires et mixtures organiques de matériaux thérapeutiquement actives. Son dispositif le plus marquant est l'emploi de tiges dans la chambre à distillation pour faciliter la formation de films de distillant.

S. W. Ferris E. R. Lamson, & D. M. Smith *U.S. Pat.* 2,514,943

Falling-Film High Vacuum Still

83/III

United States. It was proposed to purify high boiling esters and strip them from ingredients like unreacted alcohol. The esters to be treated had a molecular weight ranging from 340-460 and boiling points ranging from 230-280°C. at a pressure of 10 mm. Hg. No suitable still was available and therefore a still was specially designed for the purpose operating on the falling-film principle. The material fed into the distillation chamber is preheated to a temperature of approximately 180°C. Evaporator and condenser are arranged concentrically, the evaporator being Dowtherm-heated internally. The condenser is cooled by spraying water onto its outside circumference. The evaporator is grooved and has a total effective evaporation area of 4.3 sq. ft. The temperature in the chamber is approximately 260°C. and the pressure 0.1 to 0.5 mm. Hg. The still works satisfactorily and distills 85% of the feed on one pass with di-2-ethylhexyl sebacate as the distilland. The authors believe that the present distillation rate of 650 lbs. per hour could be raised to 2,500 lb. per hour by merely changing the dimensions of the plant. (See also abstract 84/III).

Sommaire : Description d'un appareil à distiller à film tombant et haut-vide développé par la purification d'esters à point d'ébullition élevée—(voir aussi sommaire 84/III).

Article by L. U. Spence *Industr. Engng. Chem.*, **42** Sept. 1950 1926-1929

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Remote Condenser Falling-Film High Vacuum Still

84/III

United States. The distillation unit described was developed for the processing of plasticizer-type esters (as for instance di-2-ethylhexyl sebacate). Details are given of the design of the apparatus, its operation and its performance. The still is built and operated on lines very similar to the apparatus described in abstract 83/III. However the arrangement of evaporator and condenser is different. The condenser is situated underneath the evaporator. The heat required for the evaporator is applied to the outer circumference of the same. The actual distillation chamber is used not only for distillation but also for degassing. A diffusion pump is added to the plant to provide the low pressures required for the latter process.

Sommaire : Description d'un appareil à distiller à condenseur éloigné pour usage en rapport avec le traîtement d'esters du type plastifiants—(voir aussi sommaire 83/III).

Article by F. C. Benner & A. Dinardo *Industr. Engng. Chem.*, 42 Sept. 1950 1930-1934

High-Efficiency Laboratory Fractionation. IV. A Metal Laboratory-Still

85/III

United Kingdom. An all-metal laboratory still was developed, similar to the 39 inch glass still in existence, to overcome the disadvantages caused by cleaning difficulties and breakages connected with the use of the latter. The pre-flooding arrangements and still control devices had to be modified to make the absence of visual inspection negligible. The still can be used for fractionation at pressures above or below the atmosphere.

Sommaire : Colonne de fractionnement entièrement métallique remplaçant les colonnes de verre du même type et mêmes dimensions, en vue de diminuer les bris fréquents.

Article by O. G. Dixon *J. Soc. Chem. Industr.* 69, June 1950 191-192

39 — MISCELLANEOUS PROCESSES — 39

Preparation of Carbon Targets

86/III

United States. Investigating the C^{12} (d,n) N^{13} and C^{12} (d,p) C^{13} reactions it was necessary to prepare thin pure carbon targets on light silver foils. Known methods were unsatisfactory either for the reason that the thickness of the layer could not be controlled or that the material was evaporated under bombardment. The authors developed a method of cracking benzene on silver which produced a uniform and pure carbon target. Silver foil of 3 mil. thickness was mounted on a silver disc and placed in a Vycor tube, which was sealed at one end. The open end of the tube was connected to the benzene container and to a vacuum pump. First, the tube was evacuated and then the air above the benzene removed. The benzene vapour was admitted to the tube and the tube closed. The target end of the tube was inserted into an oven, which was kept at a temperature of 1,500°F. and remained there for about 6 to 7 minutes. The thickness of the deposit depends on the amount of benzene admitted to the tube and the time the tube is kept in the oven. Details are also given of an improved method for the production of isotopically enriched C^{13} targets from methyl iodide.

Sommaire : La préparation d'anticathodes en carbone mince était nécessaire pour des recherches sur des isotopes. Une méthode de cracking du benzène sur de l'argent sous vide fut développée pour l'occasion.

Article by G. C. Phillips J. E. Richardson *Rev. Sci. Instrum.* 21, Oct. 1950 885-886

Vacuum Differential Thermal Analysis

87/III

United States. Differential thermal analysis is a technique applied to the study of certain thermal characteristics of solid complex substances. Recently this technique has become a standard analytical procedure for the investigation of complex mixtures of minerals in all such cases, where microscopic or x-ray techniques do not produce satisfactory results. However, it has been found that there are cases where this procedure cannot be applied with success on account of the combustion of the specimen. To overcome this difficulty, an apparatus was designed to carry out differential thermal analysis in a vacuum. The furnace required for the experiment is placed in a bell jar and evacuated to 1 mm. Hg with exhaustion proceeding throughout the experiment. Specimen material and reference material are placed into cups fitted to a special block in the furnace. A two-headed thermocouple is connected to each of the two cups. On the application of heat the temperature changes of the two materials are recorded for a given rate of heat supply.

Sommaire : Description d'une nouvelle technique expérimentale pour l'étude de certaines caractéristiques thermales de mélanges complexes de minéraux, dans des cas où la technique microscopique ou à rayons X n'est pas satisfaisante.

Article by W. L. Whitehead & I. A. Breger *Science*, 3 17.3.1950 279-281

Coconut Processing

88/III

United States. To make coconut acceptable as a food it requires sweetening. Before sweetening is effected the coconut meat has to be shredded and dried. In the past the sweetening was carried out in a rotary drum, where the coconut moisture content was increased from 4% (after drying) to about 12%, and the sugar was introduced in syrup form. Difficulties have been experienced mainly due to the fact that the sugar syrup tends to accumulate on the surface of the coconut shred without penetrating into the shred. The main object of this invention is to provide a sweetening process in which the sweetening agent penetrates the coconut strand properly. Roughly sugared coconut is introduced into a vessel which is quickly evacuated down to approximately 5 inches Hg. This will clear the pores of the coconut strand of air and gases. The vacuum is then broken by introducing steam into the vessel. This will cause the raw sugar to melt, spreading itself uniformly over the surface of the coconut strand and penetrate into the pores of the strand. Following this operation atmospheric pressure is admitted and the product rapidly cooled down to room temperature.

Sommaire : Il est proposé de procéder au séchage et sucrage de noix de coco sous vide.

E. F. Glabe & Food Technology Inc., *U.S. Pat.* 2,502,516

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40 — GASES AND VAPOURS — 40

Packless Valve Flow Regulator — 23/IV

See Abstract No. : 74/II

Comments on Formulae for the Flow of Gases in Pipes at Low Pressures — 24/IV

France. The author develops formulae covering the viscous and molecular flow of gases in pipes at low pressures. A simplified version of the formulae is given to meet practical requirements.

Sommaire : Développement de formules concernant l'écoulement dans une canalisation, tant dans des conditions d'écoulement visqueux que moléculaire.

Article by L. Dunoyer *Le Vide*, 5 Nov. 1950 881-886

Direct Determination of Oxygen in Compounds Containing Carbon, Hydrogen and Oxygen — 25/IV

See Abstract No. : 51/I

Mass Spectrometric Analysis of Low Concentrations of Vapours in Air — 26/IV

United States. No technique is known which is suitable for concentrating and determining contaminants in air in the range below 100 p.p.m. A procedure is described which can be usefully employed in connection with all substances which can be trapped by liquid nitrogen. Air samples were taken from the static atmosphere of a small room which was tightly closed. Mixed solvents were evaporated from a hot plate in the room and subsequently the air in the room circulated with an electric fan. The samples for the mass spectrometer were collected in previously evacuated 50 ml. bulbs fitted with stopcocks. For control purposes samples were also drawn from the same point in the room for chemical analysis. The mass spectrometer samples were attached to the inlet manifold of the instruments through a glass U-trap, cooled in liquid nitrogen. The content of the sample bulb was exhausted through the trap, the bulb sealed off and removed. The collected solvents were then vaporised into the fixed volume of the fore part of the sample system and after two minutes this volume was opened to the main expansion volume and to the leak into the spectrometer. Typical curves are given of concentration versus time of sampling for a five-component mixture and a table of the concentrations calculated from these curves. The accuracy of the method as it stands is sufficient for many studies in industrial hygiene in particular where there is a need for taking instantaneous samples.

Sommaire : Description d'un procédé concentrant et analysant avec précision des impuretés dans l'air à l'aide du spectographe de masse.

Article by G. P. Happ, D. W. Stewart & H. F. Brockmyre *Analyt. Chem.* **22**, Sept. 1950 1224-1226

Apparatus for Determining Gas Permeability of Carbon and Graphite — 27/IV

See Abstract No. : 80/II

The Action of a Gas on a Solid — 28/IV

France. A formula is developed giving the speed of the diffusion of a gas in a powder. A number of simplifying assumptions are made with regard to the consistency of the powder. Experiments were conducted to prove the formula. There was good agreement between the experimental and calculated results.

Sommaire : Recherche théorique sur la diffusion d'un gaz dans une poudre.

Article by J. M. Dunoyer *Le Vide*, 5 Nov. 1950 905-911

Determination of Heats of Adsorption Using Metal Films — 29/IV

See Abstract No. : 59/III

The Adsorption of Gases on Copper Films — 30/IV

See Abstract No. : 58/III

A Joule-Thomson Cascade Liquefier for Helium — 31/IV

See Abstract No. : 81/II

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41 — FREEZE DRYING — 41

An Investigation of High Vacuum Freeze Drying as a Means of Drug Preservation—Part I — 32/IV

United States. This is an abridged version of a report on a comparative study of the effect of drying belladonna leaves by ordinary oven and freeze drying methods. Details are given of the experimental procedure chosen and the equipment used. Leaves of Atropa Belladona L as far as possible identical in age, size, metabolic process and grown in the same enviromental conditions, were divided into two analogous samples, one of which was oven-dried and the other freeze-dried. After drying the samples were powderised and the various analyses made. The results of these experiments are given in tabulated form and a.o. the following conclusions were drawn :—(*a*) Freeze-drying of belladonna leaves is a success and should be extended to other leaves and plant organs. (*b*) Freeze-drying reduces the moisture content to the same degree as oven-drying. (*c*) The preservation of alkaloids of belladonna leaves is the same for both methods. (*d*) Oven-drying causes more proteolysis than freeze-drying. (*e*) Oven-drying tends to destroy chlorophyll while freeze-drying tends to preserve it.

Sommaire : Comparaison du séchage conventionnel de feuilles de Belladonne en étuve et le séchage par " Freeze Drying."

Article by M. A. Chambers & J. W. Nelson *J. Amer. Pharm. Ass.* 39, June 1950 323-326

Changes in the Reactivity of Purified Prothrombin After Freeze Drying — 33/IV

United States. It was observed that purified prothrombin (a protein substance present in blood plasma) could not be activated as readily as prothrombin of native plasma. This was attributed to the removal of essential activators in the purification process. Studying this closely the authors found that prothrombin preparations purified by freeze-drying methods became refractory to physiological activators such as thromboplastin. Prothrombin, thus altered, could however, be activated autocatalytically in 25% sodium citrate solution and could be recovered as thrombin (protein-like substance formed from prothrombin).

Sommaire : On étudie les propriétés et le " comportement " de la prothrombine purifiée par la dessication a froid sous vide.

Article by R. I. McClaughry E. B. Andrews & W. H. Seegers *Proc. Soc. Exper. Biol., N. Y.* 75, Oct. 1950 252-254

Process for the Desiccation of Acqueous Materials from the Frozen State — 34/IV

United States. It is proposed generally to use dielectric heating instead of contact heating in freeze-drying procedures and, in particular, to use frequencies in excess of 200 megacycles. It is added that a plant originally fitted with means for contact heating can easily be converted to h.f. dielectric heating by providing two plates in the chamber, insulated from each other and in such a position that the material to be processed can be placed between them. It is claimed that this arrangement accelerates the rate of drying by supplying the heat to the area where evaporation is needed most, *i.e.* adjacent to the ice layer. In that area, just freed from the ice, moisture content is highest.

Sommaire : Il est proposé d'amener de la chaleur à la matière congelée dans la chambre d'un Freeze Dryer à l'aide d'un champ diélectrique à haute fréquence, au delà de 200 mégacycles.

S. Bradbury & Lyophile - Cryochem. Corp. *U.S. Pat.* 2,512,991

45 — ELECTRONICS — 45

Mass Spectrometric Study of Solids. I. Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials — 35/IV

United States. Details are reported of an exploratory investigation into the usefulness of mass spectrometric techniques for the surface analysis of materials of interest to the electronics industry. A Nier-type mass spectrometer was used. The tube was made of metal and glass with copper-gasket vacuum seals. A mercury diffusion pump was used. A diagrammatic sketch is given of the double-filament ion source used. The authors studied Ni, ZnO, Mo, Cu and (BaSrCa) O surfaces but could not establish a general material transport effect completely divorced from the purely thermal effect of such bombardment. Only in one case could a specific reaction to electron bombardment be noticed, *i.e.* the ejection of O+ions from hydrogen-fired molybdenum. Thermal evaporation from cathode nickel alloys and molybdenum was studied and a copious evolution of alkali metal ions was found to take place at a moderate temperature and at higher temperatures an evolution of alkali metal atoms as well as ions.

Sommaire : Etude des effets du bombardement électronique sur des surfaces solides et les possibilités d'utiliser ces effets pour l'analyse des surfaces de matériaux par spéctrométrie de masse.

Article by R. H. Plumlee & L. P. Smith *J. Appl. Phys.* 21, Aug. 1950 811-819

The Barium Oxide on Tungsten Cathode Interfaces — 36/IV

United States. The authors studied the composition of the interface compounds formed by the reaction of oxide coatings with the base metal. Where the oxide coating on tungsten was pure BaO, the bulk of the interface compound was identified as barium tungstite, $BaWO_3$. Details of the experimental procedure are given. The cathodes were prepared by spraying $BaCO_3$ with an organic binder onto thoroughly cleaned tungsten wire

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(10 mil.). Then, the cathodes were mounted into vacuum tubes and the tubes exhausted to a pressure of 5×10^{-8}mm. Hg. A heater current was passed through the wire to reduce $BaCO_3$ to its oxide. Now, the tubes were given a life test but no emission current was drawn. After a certain period the tubes were dismantled, the oxide coatings removed from the cathodes mechanically and the exposed interfaces examined in an x-ray diffraction camera. Additional life tests were carried out to ascertain whether the thickness of the interface compound varied with time, but it was found that its thickness of 1.5×10^{-4}cm. remained the same in every case. However, drawing an emission current may have an influence on the thickness.

Sommaire : Compte-rendu sur l'existence et la composition de composés interfaciels formés par la réaction du recouvrement d'oxyde avec le métal de base dans le cas de cathodes à couche d'oxyde.

Letter by E. B. Hensley & J. H. Affleck *J. Appl. Phys.* **21**, Sept. 1950 938

The Photo-Absorption of a Thin Film of Barium 37/IV

France. The author noticed that incident light produces an appreciable change in the difference of potential between an anode of the tungsten filament type and an oxide-coated cathode. He studied this phenomenon and came to the conclusion that the incident light was ionising the barium deposited on the tungsten filament. The change of the output voltage of the anode is a result of this ionisation effect.

Sommaire : Les phénomènes d'émissions de cathodes à couche d'oxyde, causés par photo-adsorption des couches minces de barium, sont étudiés et il est tenté d'en donner une explication.

Article by Ch. Biguenet *Le Vide*, 5 July-Sept. 50 831-836

Vacuum Tubes with Mutually Bombarding Oxide Cathodes 38/IV

Australia. This is a report on the preliminary results of experiments aimed at developing a tube generating high-frequency power without the use of transformers or rectifiers when operated from an a.c. supply of no less than 240 volts. A triode tube was used consisting of two oxide cathodes, 2 mm. apart, and a grid of molybdenum wires between them. A voltage was applied to the grid in such a manner that for any half cycle it consisted of a half sine wave supplied negatively relative to the particular electrode then acting as a cathode.

Sommaire : Bref compte-rendu sur les résultats préliminaires des expériences tentant à utiliser une triode avec deux cathodes à oxyde et une grille en fil de molybdène, comme source de force haute fréquence.

Article by E. G. Hopkins *J. Appl. Phys.* **21**, Sept. 50 841-842

On the Production of Signals in Television Camera Tubes. The Conductron Tube 39/IV

France. The mechanism of signal production in different t.v. camera tubes is described, the details of a new tube, the Conductron, are given and a system of classifying the tubes according to their sensitivity is proposed.

Sommaire : Description d'un nouveau tube de caméra à télévision, le Conductron.

Article by M. W. Veith *Le Vide*, 5 Nov. 1950 887-895

47 — GLASS PROCESSING — 47

Metal Insert for Vacuum-Tight Sealing 40/IV

United Kingdom. A glass-to-metal seal is described which can be produced cheaply. Soft glass is used. The metal prongs are made of an alloy consisting of nickel (42%), chromium (4-8%), aluminium (0.12%) and iron with traces of manganese (all percentages in weight). It is essential that the alloy is kept free from cobalt and that the surface of the prong is covered with an oxide skin of at least one of the core metals. The aluminium content of the alloy is sufficient for bonding the skin to the core but insufficient to affect materially the temperature-expansion characteristics of the core.

Sommaire : Description de joints métal-verre permettant l'utilisation du verre ordinaire tendre. Il y a lieu d'utiliser un métal de composition particulière, qui est précisé dans le brevet.

Sylvania Products Inc. *Brit. Pat.* 647,257

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An. Mec. Elect.	Anales de Mecanica y Electricidad. Reina 31, Madrid, Spain.
Analyt. Chem.	Analytical Chemistry. American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Aust. J. Appl. Phys.	Australian Journal of Applied Physics. Commonwealth Scientific and Industrial Research Organisation, 314, Albert Street, East Melbourne, C.2, Victoria, Australia.
Brit. J. Appl. Phys.	British Journal of Applied Physics, The Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Canad. J. Res.	Canadian Journal of Research. National Research Council, Ottawa, Canada.
Ceramic Industr.	Ceramic Industry, Industrial Publication Inc., 5, S. Wabash Avenue, Chicago 3, Ill., U.S.A.
Chem. Age	Chemical Age, Benn Bros., Ltd. 154, Fleet Street, London, E.C.4, England.
Chem. Engng.	Chemical Engineering. McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. Industr.	Chemical Industries. McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry. Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Chemie Verlag, Weinheim/Bergstrasse, Hauptstrasse, 129, Germany.
C.R. Acad. Sci., Paris	Comptes Rendus des Séances de l'Academie des Science, Messrs. Gauthier-Villar, Quai des Grands-Augustine 55, Paris, France.
Discovery	Discovery. Jarrold & Sons, Ltd., London Street, Norwich, England.
Discuss. Faraday Soc.	Discussions of the Faraday Society, Faraday Society, Aberdeen University Press, Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Electronics	Electronics. McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Endeavour	Endeavour, 26, Dover Street, London, S.W.1, England.
Engineer	The Engineer, Morgan Bros. (Publishers), Ltd., 28, Essex Street, London, W.C.2, England.
Engng. Min. J.	Engineering and Mining Journal. McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Food Industr.	Food Industries, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Gen. Elect. Rev.	General Electrical Review, General Electric Co., 1, River Road, Schenectady 5, N.Y., U.S.A.
Il Nuovo Cimento	Il Nuovo Cimento, Nicola Zanichelli Soc. An, Via Irnerio 34, Bologna, Italy.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 33, Tothill Street, London, S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Instruments	Instruments, Instruments Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
J. Amer. Pharm. Ass.	Journal of the American Pharmaceutical Association, American Pharmaceutical Association, 2215, Constitution Avenue, N.W. Washington 7, D.C., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Iron & Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1, England.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.

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Contd.

J. Phys. Soc., Japan	Journal of the Physical Society, Japan, Faculty of Science, University of Tokyo, Japan.
J. Rech. Centr. Nat. Rech. Sci.	Journal des Recherches du Centre National de Recherche Scientifique, 13, Quai Anatole-France, Paris (VII^{E}), France.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture. Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
J. Soc. Chem. Industr.	Journal of the Society of Chemical Industry. Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Metallurgia	Metallurgia, Kennedy Press, Ltd., 31, King Street, West, Manchester 3, England.
Phys. Rev.	Physical Review, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Proc. I.R.E.	Proceedings of the Institute of Radio Engineers, Institute of Radio Engineers, 1, East 79th Street, New York 21, N.Y., U.S.A.
Proc. Phys. Soc.	Proceedings of the Physical Society, Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Soc. Exper. Biol., N. Y.	Proceedings of the Society for Experimental Biology and Medicine. College of the City of New York, New York 31, U.S.A.
PTT-Bedrijf	Het PTT-Bedrijf. Hoofdbestuur der Posterijen, Telegrafie en Telefonie, Javastraat 6, The Hague, Holland.
Rech. Aëron.	La Recherche Aeronautique, Office Nationale d'Etudes et des Recherches Aëronautiques, 3, Rue Leon Bonnat, Paris (16^{E}), France.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	Science, American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W. Washington 5, U.S.A.
Science Abstracts	Science Abstracts. The Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Taylor Technol.	Taylor Technology. Taylor Instrument Co., Rochester 1, N.Y., U.S.A.
Le Vide	Le Vide, Societe Francaise des Ingenieurs Techniciens du Vide, 44, Rue de Rennes, Paris (VI^{E}), France.
Z. Angew. Phys.	Zeitschrift for Angewandte Physik. c/o Springer-Verlag/Berlin-Charlottenburg, Jebensstrasse 1, Germany.

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